Quality Assurance Plan
of the
Chemical Division
of
DTC Laboratories, Inc.

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Table of Contents of the Quality Assurance Plan of the Chemical Division of DTC Laboratories, Inc.

1.0 Introduction

This Quality Assurance/Quality Control (QA/QC) Plan documents the the organization and plan for the assurance and control of quality in the work done by the Chemical Division of DTC Laboratory, Inc., Springfield, Illinois. It covers the guidelines on sampling, sample preservation, extraction methodology, use and maintenance of instrumentation, general laboratory support services and program, glassware and chemical documentation, laboratory personnel and data management and control in accordance with the Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans by Thomas W. Stanley and S. Sidney Verner of the U. S. Environmental Protection Agency.

The goal of this plan is to ensure that all technical data generated by the Chemical Division are technically valid, representative and legally defensible. Its primary focus is the description of those procedures which those procedures which will be used to document and report precision, accuracy, representativeness, comparability and completeness of environmental measurements.

2.0 DIVISION DESCRIPTION

The chemical division of DTC Laboratories, Inc. has as its primary function the analysis of chemicals in soil, water and other samples. Clients include state laboratories, private corporations, schools, realtors, contractors and private individuals.

The division in its commitment to quality participates in pertinent certifications and quality assurance programs, including:

- o Water Supply (WS) Quality Control Certification Program,
- o Water Pollution (WP) Quality Control Certificaton
- o Water Supply Quality Control Check
- o Water Pollution Quality Control

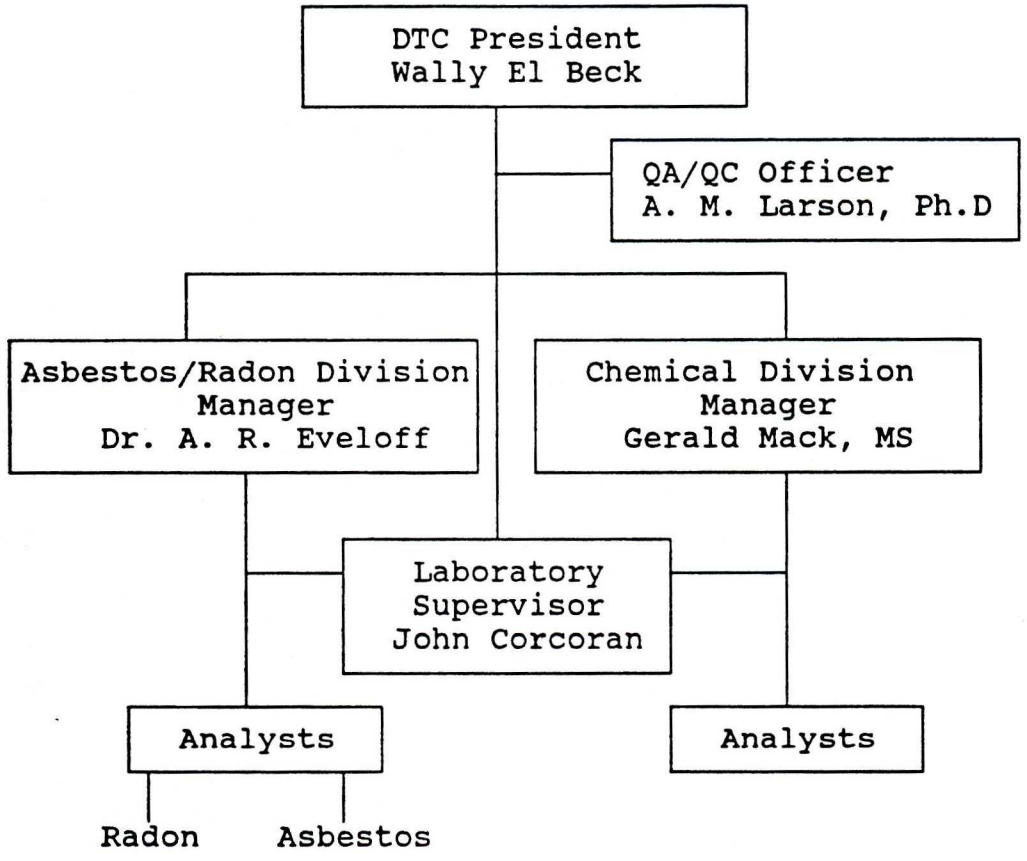
DTC Laboratory, Inc. participates in the USEPA water studies series and also utilizes internal quality control samples on a quarterly basis to insure that accuracy is maintained in analytical methodology. All solvents and reagents are regularly analyzed to ensure that there are no contamination errors.

The division has both SOPs and practiced procedures for sampling, sample custody/chain of custody procedures, each analytical procedure, recording keeping, security and quality assurance. Training manuals and analyst skill verification processes have been developed.

3.0 Division Organization and Responsibilities

DTC Laboratories, Inc., is a full service environmental testing laboratory, subdivided into two divisions; chemistry and radon/asbestos each with a manager. Each manager is responsible for the respective division and reports to the corporate president as diagramed in Figure 3.1.

Figure 3.1 Organizational Chart of DTC Laboratories, Inc.



Each division participates in external quality control programs, maintains controls and develops appropriate internal quality control measures. As an environmental testing laboratory each division manager is responsible for the development of a program that guarantees results that are accurate and precise. The division managers are responsible to the DTC Laboratory, Inc. President. The Quality Control/Quality Assurance Officer is responsible for the development and maintenance of the quality assurance program and the monitoring of the quality control programs of the two divisions. The QA/QC Officer reports directly to the president.

All samples are logged into the laboratory by the laboratory supervisor, who assigns each a number and color codes the specimens; white for chemistry, red for radon and blue for asbestos. The laboratory supervisor completes the sample custody/chain of custody forms, assigns the samples to the appropriate division and puts them in an appropriate secure area.

The analysts perform the assays and prepare the preliminary reports. All final reports are reviewed by the division manager.

Reports are reviewed by the quality assurance officer.

4.0 Personnel and Their Qualifications

4.1 Quality Control/Quality Assurance Officer

The QA/QC Officer reports directly to the Corporate President and is independent of the laboratory management organization. Duties and responsibilities include the following:

o Verifying implementation of all aspects of this QA/QC plan, including those related to sample handling, analyses, data handling and reporting, audits and corrective action.

o Effecting and maintaining effective communications on quality matters with all cognizant quality program personnel in the corporation.

o Coordination of all quality audit and overview activities.

4.2 Division Manager

Duties and responsibilities of the Division Manager include the following:

o Preparation of the procedure manuals and the precision and accuracy of the analyses.

o Directing the laboratory's analytical programs

o Ensures compliance with appropriate analytical method and instrument performance specifications

o Monitoring the facilities, the equipment, personnel, and records.

o Responsibility for the conformance of the laboratory with state and federal regulations.

Division Manager is a professional of appropriate education, training and experience who reports directly to the president.

4.3 Laboratory Supervisor

The laboratory supervisor is responsible for:

o Ensuring that all submitted samples are properly accepted into the laboratory in accordance with documented sample acceptance procedures (Section 5 and 6)

o Ensuring that samples are entered into the laboratory data management system (Section 5 and 6)

o Arranging for proper and secure sample storage.

o Coordination of projects and associated work loads

o Supervising the wet chemistry laboratory log.

The laboratory supervisor is required to have a bachelors degree in a science related field, with appropriate technical training and experience. The supervisor reports to the president and coordinates the work load of the divisions.

4.4 Analysts

Analysts' duties and responsibilities include the following:

o Equipment maintenance (Section 12.0)

o Equipment calibration (Sections 7.0)

o Sample extraction and analysis (Section 8.0)

o Raw data manipulation and reporting (Section 10.0)

o Inclusion of appropriate QC samples and considerations into all laboratory operations (Section 9.0)

Chemical analysts have appropriate formal training and experience in the tests each performs. Each analyst has a resume which contains a summary of training and experience with copies or certification on file. Each analyst knows and takes the necessary personal sanitation and health precautions designed to avoid contamination of self and test.

Each analysts receives formal instruction and goes through an apprenticeship period under supervision of the division manager and laboratory supervisor. The ability of an analyst to do each test is verified by the division manager. A minimum of two years of college chemistry is required with a bachelors degree in an appropriate science related field preferred.

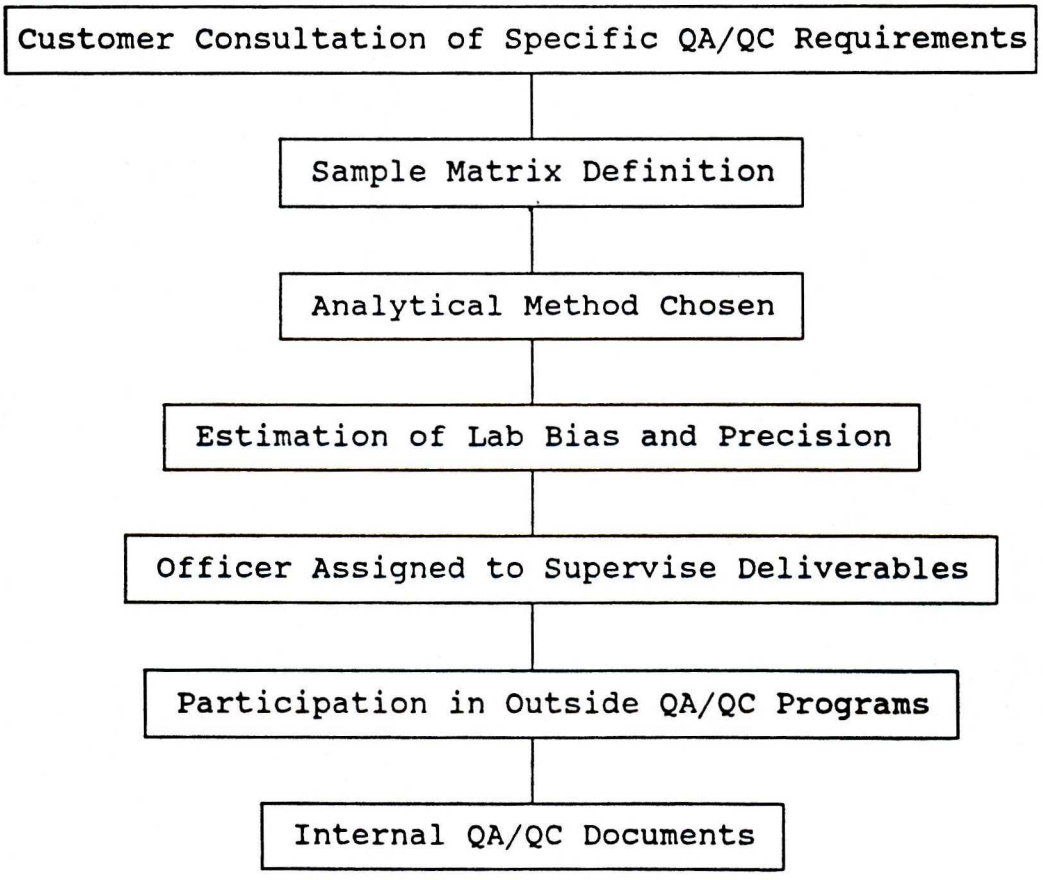
Analysts report to their respective Division Manager.

5.0 Quality Assurance Objectives for Measurement in terms of Precision, Accuracy, and Completeness.

At DTC Laboratories, Inc. quality assurance involves the documentation of the step-by-step rigorous control of sample analysis parameters that insure that results are accurate and defensible. Quality assurance involves the minute details of sampling, sample preservation, extraction methodology, use and maintenance or instrumentation, general laboratory support services and programs, glassware and chemical documentation, laboratory personnel and data management and control.

Quality control is the natural complement of quality assurance that focuses on the utilization of statistical models to estimate within a degree of confidence the accuracy and reproducibility of a particular data set. The Quality Control Decision Chart (Figure 5 - 1) is used to determine the specific quality control matrix.

Figure 5 - 1 DTC Laboratory's Quality Control Decision Chart



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DTC Laboratories methodologies are based on those mandated by the United States Environmental Protection Agency in the publications "Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/14-79-19, March 1979. etc.) All measurements are representative of the media and conditions being measurements. All data is calculated and reported in units consistent with those in the industry.

The object the QA/QC program is to define the quality goals on data generated during the chemical analysis and assure accuracy, precision, representativeness, comparability and legally defensible results.

5.1 Definition of Terms

ACCURACY - The difference between the mean of a set of results and the true or correct value. The absolute accuracy is the difference between the mean and the true value, and the relative accuracy is the absolute accuracy divided by the true value. Relative accuracy is commonly expressed as percent accuracy.

PRECISION - The reproducibility of measurements within a set expressed as percent variance from a true value, or when the true value is unknown the degree of mutual agreement among individual measurements is expressed by the Relative Percent Difference, RPD.

REPRESENTATIVENESS - The degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition.

COMPARABILITY - The confidence with which one data set can be compared to another.

5.2 Procedures of QA/QC Standards Development

DTC Industries, Inc. maintains an internal QA/QC standard to guarantee precision, reproducibility and accuracy and its verification. DTC works with the client to determine the degree of precision needed for the specific type of sample being tested. This joint decision is based on:

1. Sample Matrix
2 Analysis Requested
3. Methodology and Instrumentation Required

From these factors, a consensus on available and needed quality can be determined. Estimates of confidence takes into account all the variance arising from processes performed to prepare the sample for analyses, variation within the analytical technique and includes the desires and requirements specified by the client. The results of all blanks and other controls that apply to their samples are provided to each customer.

DTC Laboratories believes that it is better to approach "accuracy" as the total error of a particular result which is the combined random and procedural errors rather than to consider only the procedural errors as is typically done.

Unless the client requires a different level of accuracy, DTC Laboratory will implement the accuracy, DTC Laboratory will implement the following schedule:

1. The test bias of appropriate samples shall not exceed 15 % of the measured value or 1/2 of the required method detection limit, whichever is greater.

2. The analytical result's total error shall not be greater than 30% of the results or the required method detection limit, whichever is greater.

3. The standard deviation of the results as defined by

$$SD = \frac{N \sum (X)^2 - \sum (X^2)}{N(N-1)}$$

shall not exceed 25 % of the required detection level of 7 % of the measured value, whichever is greater.

5.3 Objectives Defined

The accuracy, precision, and completeness objectives are summarized in TABLE 5 - 1. The accuracy and precision control objectives are derived from EPA method control limits published with each analytical method (TABLE 5 - 2) The listed objectives are representative for the sample matrices and conditions usually tested.

The accuracy and precision measurements will be monitored by comparing the results of each spike with spike duplicate recovery to the appropriate objective. The quality control chart for each parameter will be reviewed to assure the completeness objective.

Representativeness will be achieved by the random selection of samples which characterize the sample matrix. Observable parameter variation, matrix differences and environmental conditions will be noted.

TABLE 5-1 - ACCURACY. PRECISION. AND COMPLETENESS OBJECTIVES FOR MEASUREMENT DATA

Parameter	Accuracy %	Precision	% Completeness
PCBs			
PCB-1016	50-120	20	95 %
PCB-1221	15-178	33	95 %
PCB-1232	10-215	35	95 %
PCB-1242	39-150	21	95 %
PCB-1248	38-158	24	95 %
PCB-1254	29-131	20	95 %
PCB 1260	8-127	22	95 %
TOC	53-129(1)	20(1)	95 %(1)
TOX	55-129(1)	20(1)	95 %(1)
Organo Chlorine	NA	20	95 %
BTU/lb	NA	20	95 %
METALS			
Antimony	66-120	20	95 %
Arsenic	61-137	20	95 %
Barium	75-120	20	95 %
Beryllium	77-120	20	95 %
Cadmium	80-120	20	95 %
Chromium	65-125	20	95 %
Copper	80-120	20	95 %
Lead	72-125	20	95 %
Mercury	80-120	20	95 %
Nickel	80-120	20	95 %
Selenium	80-120	20	95 %
Silver	76-120	20	95 %
Thallium	62-129	20	95 %
Zinc	80-120	20	95 %
INORGANIC NON-METALLICS			
Chloride	80-120	20	95 %
pH	NA	NA	95 %
Cyanide	69-120	20	95 %
Sulfide	80-120	20	95 %

TABLE 5-1 - ACCURACY. PRECISION AND COMPLETENESS OBJECTIVES FOR MEASUREMENT DATA (CONTINUED)

Parameter	Accuracy %	Precision %	Completeness
PHYSICAL PROPERTIES			
Moisture Content	NA	20	95 %
Solids, Total	NA	20	95 %
RCRA ANALYSIS			
Ignitability	NA	NA	95 %
Reactivity Cyanide	69-120	20	95 %
Reactionary Sulfide	69-119	20	95 %
EP Toxicity (for metals only)	NA	NA	95 %

(1) These objectives applicable to a water matrix only.

NOTE: All objectives unless otherwise noted are applicable to all sample matrices.

6.0 Sampling Procedures and Containers

DTC Laboratories, Inc. is primarily a testing laboratory. Included in the analytical services provided, sample containers are supplied with complete instructions for the collection and transport of the sample. (Figure 6 - 1) Shipping packs are provided when applicable.

6.1 Sample Containers

Sample containers will be purchased, precleaned, from a contractor who prepares sample containers in accordance with EPA p/ocedures. If appropriate containers are not available, vials will be cleaned according to EPA specifications.

Each sample container is lot coded at DTC Laboratories, and the documentation as to its suitability for the transport of analytical samples is maintained here (Figure 6 - 2) Copies of complete documentation on applicable sample containers is available upon customer request.

All sample containers are provided with a label which provides container reference information as well as room for customer identification. (Figure 6 - 3) Appropriate field blanks, laboratory prepared and sealed, are included with each shipment. Sample information sheets and sampling instructions are always included.

6.2 Sample Collection Procedures

In those circumstances in which a DTC analyst collects samples, USEPA-approved collection methods are employed.

Figure 6 - 1 Description for Containers

Sample	Parameter to be tested	Method	Type of Container
Soil	Acid/base/Neutral + Pesticides + PCBs + Dioxin/Furan Scan	2270	3 oz wide mouth glass jar with Teflon liner
Ground water	Acid/base Neutral + Pesticides + PCBs + Dioxin/Furan Scan	2270	1 gal amber glass bottle with Teflon liner
Freon Extract	Benzo(a)pyrene, naphthalene, Fluoranthene, Anthracene, Pyrene	2270	3 oz wide mouth glass jar with Teflon liner
Soil	Volatile Organic, Priority Pollutants	2740	4 oz wide mouth glass jar with Teflon liner
Ground water	Volatile Organic Priority Pollutants	2740	2-4 ml glass vials with Teflon septa
Freon Extract	Oil and Grease	9070	In same containers as Freon extract
Soil	Antimony	7040	1 gal glass or plastic jar *
Soil	Arsenic	7060	1 gal glass or plastic jar *
Soil	Beryllium	7090	1 gal glass or plastic jar *
Soil	Cadmium	7130	1 gal glass or plastic jar *
Soil	Chromium	7190	1 gal glass or plastic jar *
Soil	Copper	7210	1 gal glass or plastic jar *
Soil	Lead	7420	1 gal glass or plastic jar *
Soil	Mercury	7470	1 gal glass or plastic jar *
Soil	Nickel	7520	1 gal glass or plastic jar *

Sample	Parameter to be tested	Method	Type of Container
Soil	Selenium	7740	1 gal glass or plastic jar *
Soil	Silver	7760	1 gal glass or plastic jar *
Soil	Thallium	7840	1 gal glass or plastic jar *
Soil	Zinc	7950	1 gal glass or plastic jar *

(1) Will use Method 82BD for Dioxin/Furan Scan
Willl use Method 6DB for Pesticide/PCB's analysis

(2) Will use Method 6D1D? (ICAF)

* 1 sample for all soil metals

Figure 6 - 3 Sample Identification Form

DTC Laboratories, Inc.
(217) 529 - 9191

Sample Identification Document
Returned by Customer

Client:_____		
_____		
_____		
Project Number	Code	
Source Location Sampled		
Date	Time	AM PM
Container QC Code	Data Location	
Customer QC		
Container Preparation Date	Method	
Preparers Signature		
Analysis Requested		

7.0 Chain-of-Custody

At DTC Laboratories, chain-of-custody is an all-pervasive concept. At any time from the moment the sample bottle is either sealed by our analysts or received in the laboratory until the last extraction is finished, all of the sample, its extracts, and/or fractions are documented. The initial control of the sample is at time of collection and from that moment there is a documented paper trail to define precisely who had control of the sample, what processes were performed, when they were done, who did them and where the results can be located. A sample chain-of-custody form is shown in Figure 7 - 1.

7.1 Chain of Custody Procedure

The designated Laboratory Sample Custodian will receive and document all sample submittals into the laboratory in the following manner.

- o Examine the condition, preservation and accompanying documentation of all submitted samples prior to approval and formal acceptance into the laboratory.
- o Resolve any sample condition, preservation or documentation discrepancies before the sample is approved and accepted into the laboratory.
- o Record all required acceptance data in the laboratory sample log in the approved format.
- o Document the accuracy of the data by reviewing the entries and the submittal data.
- o Label each sample with a laboratory sample identification number
- o Place the samples in a secure sample storage area for distribution to the appropriate analyst.

Each sample shall be tracked from the time it is collected or received with sampling form/chain of custody form, accession numbers applied and standard tracking form. Multiple site samples collected by DTC Laboratories, Inc. will also have a Field Tracking Report

7.1 Security

Samples shall be under the control of the laboratory supervisor or designee from the time of acceptance until final deposition.

Figure 7 - 2

Chain-of-Custody Document

Client:_____

Date Received:_____

Sample Number(s):_____

SAMPLE INFORMATION

Sample Type: Soil_____ Water_____ Sludge_____
Radon_____ Asbestos_____
Other_____

Sampling Done By: Client_____ DTC_____

Samplers Name_____

Method of Receipt: ___ Mail ___UPS____FedEx ___Hand Carry
Other:

Receiving Officer: (print name)_____

Sample Logged-In By: _____

ANALYSIS REQUESTED

	Hazardous Waste Stream Analysis with Pesticides	_____	Hydrocarbons
	Hazardous Waste Stream Analysis w/o Pesticides	_____	Pesticides
	Non-Hazardous Waste Stream Analysis with Pesticides	_____	Volatiles
	Non-Hazardous Waste Stream Analysis w/o Pesticides	_____	PCBs
	Other	_____	Dioxin
		_____	Total Heavy Metals
		_____	Other_____
		_____	Radon
		_____	Asbestos - bulk
		_____	Asbestos - air
		_____	
		_____	
		_____	

Comments:_____

_____

_____

_____

Figure 7 - 3

Record of Sample Location

Original Container Type__________________ Sample Number

TRACKING

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

Sample Extracted for ____________________________________
Extracted by ______________________ Date:________________
Amount of Sample Used ____________
Final Amount of Sample ____________
Extract Stored at _________________ in __________________

8.0 Calibration Procedures and Frequency

Specific procedures for the operation and calibration of all analytical instruments will be maintained by the DTC Laboratory, Inc. Along with proper maintenance, these procedures ensure optimum instrument performance and accuracy. These procedures include: proper operator training and supervision; mandatory instrument performance specifications; and systematic instrument calibration, verification and monitoring schedules.

8.1 Laboratory Instruments

The laboratory utilizes mandatory instrument performance specifications to constantly ensure optimum instrumental performance. These performance specifications require acceptable instrument response to specific performance standards prior to initiating further instrument calibration and analyses. Acceptable instrument response criteria are based upon the manufacturer's or EPA's analytical specifications.

Laboratory analysts record and document all instrumental runs in designated laboratory instrument logbooks (Section 10.2.2). These logbooks identify instrument operating parameters, settings, and performance data associated with each instrumental run. Instrumental runs pursuant to establishing instrument performance specifications and calibration are also recorded in these laboratory instrument logbooks.

The laboratory utilizes systematic instrument calibration procedures to constantly ensure analytical accuracy. Initial instrument calibration curves are generated, systematically verified, and routinely monitored throughout the duration of all instrumental analyses. Initial multipoint GC and GC/MS calibration curves are verified by injection of a known working standard every twelve (12) hours of instrument operations. An unacceptable working standard response (15% variation for Method 8080, 10% for Method 6010, and 20% for other methods) requires construction of a new calibration curve. Other instrument calibration curves are constructed immediately prior to analyses of each batch of samples. All initial instrument calibrations and/or verifications are then routinely monitored by systematic, repeat injections of appropriate check standards throughout the duration of analyses. Additionally, all GC/MS analyses will utilize internal standards to ensure high quality data.

Specific calibration procedures for laboratory instruments including the frequency and standards used are listed in Table 7-1. Table 7-2 details the equipment be used in the laboratory.

8.2 Measurement Equipment, Glassware, Water, Reagents. and Industrial Gases

The laboratory will adhere to proper standards of good laboratory practice in utilizing measuring equipment, glassware, water, chemical reagents, and industrial gases. Adherence to proper standards relating to these laboratory elements will validate analytical data. All laboratory glassware, balances, thermometers and subsequent volume, mass, and temperature measurements are directly traceable to primary standards. Chemical reagents and industrial gases are purchased and utilized as appropriate for various laboratory applications.

- o Volumetric Glassware will conform to National Bureau of Standards (NBS) Class A standards. All mechanical pipettes are calibrated annually with the MLS pipette volume calibration kit. All calibrations are recorded and documented in laboratory calibration logbooks. Written procedures (SOPs) for cleaning and storing glassware will be posted at appropriate wash stations.

- o Laboratory balances are annually serviced and calibrated under the manufacturer's service contract. Additional balance performance evaluations are conducted routinely by comparison against NBS certified weights. Unacceptable performance require manufacturer's service adjustments. Both balance service and performance evaluations are recorded and documented in laboratory equipment performance logbooks.

- o Laboratory and field thermometers are calibrated against a NBS certified thermometer and recorded in the designated laboratory thermometer calibration logbook. Laboratory drying ovens, incubators, refrigerators, etc., contain calibrated thermometers. Temperature readings are recorded daily in laboratory temperature logbooks. Unacceptable deviation from desired temperatures require immediate corrective action.

o Laboratory pure water is generated by a commercial on-line water purification system consisting of mixed resin deionizing and carbon filtration cartridges. Cartridges are routinely replaced and serviced by manufacturer or as indicated by an on-line resistivity indicator or laboratory method blank contamination. All water purity information is recorded in the manufacturer's service file.

o The laboratory will utilize various types and purities of chemical reagents, solvents, and industrial gases depending upon their intended use. Laboratory stock and working standards are derived from commercially available primary standards and solvents whenever possible. These stock and working standards are properly labeled (content, concentration, date, analyst) and routinely checked for degradation and/or impurities in accordance with the appropriate analytical method specifications. On-line molecular sieves and oxygen traps are used where appropriate to remove impurities from desired industrial gases. All chemical reagents, solvents, and industrial gases are stored only in designated areas in accordance with the Laboratory Health and Safety Program.

Table 8 - 1 LABORATORY INSTRUMENT CALIBRATION PROCEDURES

GC/MS

Every 12 hours, the instrument shall be tuned to DFTPP or BFB to assure that the instument response meets EPA specifications.

Generation of five (5) point calibration curve for all method compounds monthly, or more frequently if needed.

Verification of system cleanliness by the analysis of a reagent blank during every 12 hour tune.

Analysis of a standard after the analyses of 10 sample to assure the consistency of instrument response.

Addition of internal standards to each sample

GC

Generation of five (5) point calibration curves for all analyzed compounds monthly or prior to any sample analysis, as needed.

Monitor consistency of instrument response through the analysis of a standard after every ten (10) sample analyses.

Method 8080 requires a Relative Standard Deviation (RSD) acceptance criterion of 20% for calibration factors.

Demonstrate system cleanliness through the analysis of a reagent blank prior to any sample analysis.

Maintain sample response within the limits of the response of the standards.

AA ICP

Construction of a three (3) point calibration curve for an element prior to the analysis of any sample set.

Verification of the cleanliness of the system for each element through the analyses of a reagent blank with any analytical set.

Verification of instrumental consistency through the analysis of a calibration blank and a single point check standard, after analysis of every ten (10) samples.

Bracketing of a sample response between the range of standards response.

Before beginning a sample run, the zero blank is run to determine the signal to noise ratio. (MDL/2)

pH and Ion-Selective Electrodes

Construction of a three (3) point (2 pH for pH) calibration curve weekly or prior to the analyses of any sample.

Bracketing of sample response within the limits of standards response.

Verification of cleanliness of the analytical system through the analysis of a reagent blank.

Verification of instrument consistency through the analysis of standards after the analysis of every twenty (20) samples (where applicable).

Spectrophotometer

Construction of a four (4) point calibration curve plus a blank, prior to the analysis of any sample.

Monitor for the introduction of any interferant through the analysis of a reagent blank prior to any sample analysis.

Bracket sample response within the standards response.

Verification of the consistency of instrument response through the analysis of a standard after every twenty (20) sample

8.3 ANALYTICAL PROCEDURES

8.3.1 Sample Receipt

See Section 6.2. The Action Flow Charts of Water and Solids can be found in Figures 8 -3 and 8 - 4.

8.3.2 Sample Preparation

All samples are prepared in accordance with the methods outlined in Table 8-1.

8.3.3 Equipment Start-up and Performance Check

See Section 9.2.1.1.

8.3.4 Detection Limits

A listing of all applicable analytical detection limits (DL) for this project is contained in Table 8-2.

8.3.5 Initial and Continuous Calibration

See Sections 9.2.1.1, 9.2.1.2 and 9.2.1.4.

8.3.6 Analytical Methods

A summary of all Laboratory analytical methods utilized for the LaSalle Electrical Utilities PCB Abatement Project is outlined in Table 8-1.

8.4 Analyses of QC Samples

See Sections 9.2.1.2 and 9.2.1.3).

TABLE 8-1 - LABORATORY ANALYTICAL METHODS SUMMARY

Parameter	Methods	
	Water (and non-aqueous liquids)	Solids
ORGANICS		
PCBs (Site) level1)/8080	SW846 3510/8080	SW846 3550 (med.
PCBs (HQ)	SW846 3510/8080	SW846 3550/8080
TOC	SW846 9060	NA
Organo Chlorine	NA	ASTM 2361-85/D808-81
BTU/lb	NA	ASTM D240-85/D2015-85
METALS		
Antimony	SW846 6010	SW846 6010
Arsenic	SW846 7060	SW846 6010
Barium	SW846 6010	SW846 6010
Beryllium	SW846 6010	SW846 6010
Cadmium	SW846 6010	SW846 6010
Chromium	SW846 6010	SW846 6010
Copper	SW846 6010	SW846 6010
Lead	SW846 6010	SW846 6010
Mercury	SW846 7470	SW846 7471
Nickel	SW846 6010	SW846 6010
Selenium	SW846 7740	SW846 7740
Silver	SW846 6010	SW846 6010
Thallium	SW846 6010	SW846 6010
Zinc	SW846 6010	SW846 6010
INORGANIC PNEUMATICS		
Chloride	EPA 325.3	NA
pH	EPA 150.1	SW846 9045
Cyanide	EPA 33 5.2	9010
Sulfide	EPA 376.1	9030
PHYSICAL PROPERTIES		
Solids, Total	EPA 160.3	ASTM D2109-85
Moisture Content	NA	ASTM D2216-80

Parameter	Methods	
	Liquid (and non-aqueous liquids)	Solids
RCRA ANALYSIS		
Corrosiveness	SW846 1110	SW846 9045
Ignitability	SW846 1010	SW846 1010
Reactivity SW846 9030 (7.3.4)	9010, 9030	SW846 9010
EP Toxicity	SW846 1310	SW846 1310

REFERENCES:

1. EPA - Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March, 1983.

2. SW846 - Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, EPA, September, 1986

3. ASTM - American Society for Testing and Materials (ASTM) Annual, Revised, 1987.

TABLE 8-2 - LABORATORY DETECTION LIMITS SUMMARY

Parameter	Methods	
	Water (and non-aqueous waste)	Solids
ORGANICS		
PCBs	1 µg/L	.5 mg/kg
TOC	10 mg/L	NA
Organo Chlorine	-----	.05 %
BTU		1000 BTU/lb
METALS		
Arsenic	0.005 mg/L	0.3 mg/kg
Antimony	0.2 mg/L	4.0 mg/kg
Barium	0.05 mg/L	0.03 mg/kg
Cadmium	0.01 mg/L	0.02 mg/kg
Calcium	0.05 mg/L	
Chromium	0.02 mg/L	0.04 mg/kg
Copper	0.01 mg/L	0.03 mg/kg
Iron	0.06 mg/L	0.10 mg/kg
Lead	0.05 mg/L	0.3 mg/kg
Magnesium	0.05 mg/L	
Manganese		0.01 mg/kg
Mercury	0.005 mg/L	0.15 mg/kg
Nickel	0.04 mg/L	0.03 mg/kg
Phosphorus	0.15 mg/L	0.30 mg/kg
Selenium	0.005 mg/L	0.03 mg/kg
Silver	0.01 mg/L	0.2 mg/kg
Thallium	0.1 mg/L	2.0 mg/kg
Zinc	0.01 mg/L	0.02 mg/kg
INORGANICS NONMETALLICS		
Chloride	2.0 mg/L	
pH		
Cyanide	.005 mg/L	1.0 mg/kg
Sulfide	1.0 mg/L	50.0 mg/kg
PHYSICAL PROPERTIES		
Ash Content	.5 %	.5 %
Solids, Total	5 mg/L	.5 %

TABLE 8-2 - LABORATORY DETECTION LIMITS SUMMARY (CONTINUED)

Parameter (and non-aqueous waste)	Water	Solids
Moisture Content		.5%
RCRA ANALYSIS		
Corrosiveness	.5mm per/yr	
Ignitability		
Reactivity		
Cyanide	.01 mg/L	1.0 mg/kg
Sulfide	2.0 mg/L	50.0 mg/kg
EP Toxicity (see individual metals)		

9.0 Laboratory Internal Quality Control Checks

Analytical quality control checks are performed regularly using the procedures based upon USEPA analytical methods guidance and generally accepted standards of good laboratory practice as outlined in Table 9-1. Key components of the laboratory analytical quality control program include the following quality control practices and considerations:

- o designation of laboratory Quality Control Manager to implement the laboratory QA/QC Program (Section 4.1).

- o adherence to specified laboratory sample acceptance procedures to ensure proper handling, processing, and storage of submitted samples (Section 7.0).

- o utilization of USEPA-approved analytical methods and instrumentation (Section 8.0)

- o adherence to mandatory procedures for operation, calibration, and maintenance of laboratory instrumentation (Sections 7.0 and 12.0).

- o utilization of proper laboratory measuring equipment, glassware, water, chemical reagents, industrial gases (Section 7.3).

- o constant surveillance and documentation of acceptable analytical method accuracy and precision through initial analytical method performance evaluations (Section 9.1.1) and matrix spike/spike duplicate evaluations (Sections 9.1.2).

- o utilization of continuos surrogate spike recovery evaluations where appropriate to ensure acceptable method performance (Section 9.1.4).

- o utilization of systematic method blank evaluations to identify analytical system interferences and background contamination levels (Section 9.1.3).

- o adherence to proper laboratory documentation measures to ensure the complete integrity and legal validity of all laboratory analyses (Section 10.2).

- o utilization of voluntary intra-laboratory performance evaluations to internally assess and evaluate analytical performance (Section 11.0).

o participation in numerous laboratory certifications, audits, and approval programs (Section 11.0).

9.1 Data Quality

The principle criteria for validating data quality is the continuous monitoring of acceptable analytical accuracy, precision, and overall method performance through systematic analysis of quality control samples. The laboratory will conduct both initial and continuous analytical method performance evaluations to ensure that all generated analytical data meet acceptable quality control method performance criteria established by the USEPA and the laboratory. Each analytical method commonly used in the laboratory utilizes specific quality control procedures to continually monitor acceptable analytical method accuracy and precision. These method quality control procedures primarily involve the mandatory systematic insertion of quality control samples into 10% of all laboratory analyses, (or 1 QC sample per analytical run, whichever is greater) in addition to strict adherence to instrumental performance and calibration specifications. These specific quality control procedures are thoroughly detailed in the USEPA and ASTM Methods listed in Table 8-1, and are incorporated herein by reference.

9.1.1 Initial Method Performance Evaluation

Prior to the analysis of any samples, the laboratory conducts initial method performance evaluations of each analytical method commonly used in the laboratory to demonstrate the ability to achieve acceptable method accuracy and precision. Quality control data derived from these evaluations is compared to published USEPA method performance criteria in order to determine and document acceptable laboratory analytical method capability. This initial method performance evaluation is conducted as summarized below.

9.1.1.1 Gas Chromatography/Mass Spectroscopy

A minimum of four (4) matrix spike samples are prepared using the particular

representative sample matrix and analytical parameter(s) of interest.

These matrix spike samples are prepared such that the parameter concentration(s) are within the working range of the method and at least two (2) times greater than the method's background level.

The matrix spike samples are analyzed in accordance with the method, and the average percent recovery (R) and the standard deviation of the percent recoveries (s) will be calculated from the analytical results. The laboratory values of R and s are compared to the published EPA method performance values of average recovery (X) and standard deviation (p) (if applicable). The laboratory values are not acceptable if $s > 2p$ or $(X-R) > 2p$. Unacceptable values require the laboratory to review potential analytical problems and repeat the initial method performance evaluation until acceptable values are obtained.

The laboratory does not analyze any actual samples until the initial method performance evaluation demonstrates acceptable laboratory analytical method capability. Results from the initial method performance evaluation are entered into the analytical method's initial accuracy and performance record maintained in the laboratory QC manual.

9.1.1.2 Matrix Spike/Spike Duplicate Evaluations

The laboratory conducts continuous method performance evaluations for each analytical method commonly used in the laboratory to continuously demonstrate and document the maintenance of acceptable method accuracy and precision. Laboratory quality control charts are constructed from this data in order to monitor and compare actual laboratory quality control data with acceptable published USEPA or laboratory method performance criteria.

Quality control samples consist of method blanks and matrix spike/spike duplicate samples.

Mandatory matrix spike/spike duplicate samplesare systematically analyzed in order to maintain continuous surveillance of acceptable method performance. Approximately sixty-seven percent (67%) of all quality control samples are matrix spike samples. Percent recovery determinations (R) from these matrix spike results are monitored to provide a continuous measure of the overall accuracy and precision of the method in addition to determining extraction efficiencies and sample matrix effects (see Section 13.3). For this project, spiking compounds and concentration levels will be determined based on parameters found in samples from the site. Normal spiking levels will be at ten times (10X) detection limits.

9.1.1.3 ICP

The ICAP interference check sample must be within ± 2 standard deviations from the mean value. Table 9 - 2.

Table 9 - 2 ICP Interference Check Sample Concentrations

Analyte	(mg/l)	Interferents	(mg/l)
Ba	0.5	Al	500
Be	0.5	Ca	500
Cd	1.0	Fe	200
Co	0.5	Mg	500
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

9.2 Method Blank Evaluations

The laboratory will systematically prepare and analyze method blanks to continuously evaluate analytical system interferences and background contamination levels. Method blank analyses include all components (glassware, chemical reagents, environment, etc.) of actual, routine method analyses, substituting reagent water or other applicable clean matrix for the actual sample. Approximately thirty-three percent (33%) of all quality control samples are method blanks. Analyses of method blanks will provide a safeguard against interfering and/or contaminated reagents, glassware, and laboratory environments. The results of all method blank analyses will be recorded in the Laboratory Computer Data Management System. Unfavorable method blank performance will render associated data suspect and require corrective action (see Section 14.2).

9.2 Continuous Surrogate Spike Recovery Evaluation

The laboratory will conduct continuous surrogate spike recovery evaluations on all GC/MS volatile and semivolatile analyses to ensure acceptable method performance. Surrogate spikes consisting of similar, non-method compounds and/or method compound analogues are added to each GC/MS volatile and semivolatile fraction to continuously evaluate and document acceptable method performance, without interfering with actual method compounds.

Surrogate spike recoveries must compare favorably to published USEPA methods or statistically derived laboratory performance limits in order for an analysis to be acceptable. Unfavorable surrogate spike recoveries render associated data suspect and require corrective action (see Section 14.2). For Method 8080, di-butylchlorendate and 2,4,5,6-tetrachloro-meta-xylene will be used as surrogates, as long as the compounds are commercially available.

10.0 Data Reduction, Validation, and Reporting

Raw data collection and calculations are done by our analytical personnel. Interpretation of these results will be prepared and provided to the customer complete with all appropriate quality-control information pertinent to the scope of the analytical project. All analytical observations and/or comments by our personnel will be included.

All documents produced for a single sample analysis including pertinent quality control information shall be archived at DTC Laboratory to provide a permanent record of these analyses.

10.1 Data Reduction Methods

10.1.1 Data Management System

The laboratory has developed a process that allows us to record, document, and assimilate pertinent laboratory technical and administrative data. This process provides data management functions for a number of component laboratory activities including: laboratory sample acceptance, sample analytical results, sample status and tracking, analytical QA/QC, and final report generation. The system is summarized below.

o An individual laboratory identification number is assigned to each sample and pertinent technical and administrative including sample identification, sample physical description, sampling date, required analytical parameters and required completion date.

o The above data is assimilated and a laboratory work sheet is generated. These worksheets identify the appropriate analytical parameters and associated methods necessary to complete the requisition sample analyses along with the turnaround time requirements. These turnaround requirement not only specify completion dates, but identify maximum allowable holding times for samples and/or extracts prior to analyses.

o Laboratory personnel maintain the analytical results while the associated QA data is evaluated by the QA\QC Officer.

10.1.2 Data Validation

The principle criteria to be used to validate data integrity during collection will be the following:

- o Reagent blank results
- o Method preparation blank results - Calibration verification
- o Matrix spike/spike duplicate results
- o Quality Control check sample results

These measurements are made by the analyst, using specific criteria. The analyst will either proceed with the analyses or take corrective action (Reference Section 14.00) All QC data is reviewed by the Division Manager to ensure that all QA procedures have been completed. In addition, 10 % of all raw data will be reviewed by the group leader to ensure that the method was in control during the analytical run.

10.1.3 Data Validation Documentation

The laboratory will utilize complete documentation measures to ensure the integrity and legal validity of all sample analytical results. These documentation measures encompass all analytical activities to create a traceable, legal history of each sample and subsequent analysis. All commented information is recorded in bound, consecutively-numbered analytical log books.

10.1.4 Logbooks and Written Records

DTC Laboratories follows the guidelines of the American Society for Testing and Materials E899-82 "Standard Guide for Records Management in Mass Spectrometry Laboratories Performing Analysis in Support of Nonclinical Laboratory Studies. Logbooks are bound, numbered consecutively with places for headings and signatures. All entries will be done in black ballpoint pens that meet the performance standard in Federal Specification GG-B-60D section 3.5(5).

10.1.4.1 Laboratory Sample Logbook

Sample submitted for laboratory analysis are recorded and documented in the laboratory sample logbook. Individual log entries include client, laboratory sample identification number, sample description, analytical requests, chain of custody possession statements and additional information.

10.1.4.2 Laboratory Method Logbook

All laboratory analyses are entered into various laboratory method logbooks. Each laboratory area has a logbook which records pertinent preparation, extraction and instrumental data for each sample. This includes laboratory identification number, initial sample volume or weight, extraction volumes dilution factors, values, and the name of the analyst. DTC Laboratories requires that all data generated during the conduct of an assay be recorded promptly and legibly in ink. All entries will be dated and signed or initialed by the analyst entering the data. The general format for Laboratory Logbook entries may be found in Figure 10 - 2.

10.1.4.3 Laboratory Instrument Logbooks

All laboratory analyses requiring analytical instrumentation are recorded in various instrument log books which categorically record and document analytical instrument settings and performance data. These log books record instrument calibration data, specific sample volumes, instruments parameters, and corresponding performance data for each sample. Instrumental information has been included and combined with laboratory logbooks whenever possible to consolidate data.

10.1.4.4 Laboratory Instrument Service Logbook

The maintenance, repair, adjustment and service of all instruments is recorded in appropriate service log books.

10.1.4.5 Laboratory Equipment Calibration Logbooks

All Laboratory measuring equipment calibrations are recorded in various laboratory calibration logbooks. These log books record the dates and primary standard for calibration of various laboratory thermometers, balances, and glassware items.

10.1.4.6 Laboratory Chromatography Data File

All chromatography data is categorically filed in the laboratory chromatography data file. The files include labeled, numbered chromatograph with corresponding integrator printouts and raw data sheets (Figure 10 - 2, a and b)

Figure 10 - 1 Sample Logbook Entry Organization

(a) EP TOX Digestion Setup

Title_____ Date _____

Sample Number	Sample Size	Into Water	Total Volume

Signature_____ Date _____
Verification _____ Date _____

(b) Flash Point

Title_____ Date _____

Sample Number	Dilution	TOC	DT/ΔEV	Calculations

Signature_____ Date _____
Verification _____ Date _____

(c) PNA

Sample Number	Injection	Sample Number	Injection

Signature_____ Date _____
Verification _____ Date _____

10.1.4.7. Laboratory GC/MS Data File

All Chromatography data and corresponding quantization lists generated by the laboratory GC/MS systems are categorically filed in the laboratory GC/MS data file. The file is combined with appropriate Laboratory chromatography data files for GC data. Processed GC/MS data is filed on a daily basis in the appropriate laboratory processed file. In addition, all GC/MS chromatography data, quantization lists and process data are recorded on magnetic media.

10.2 Data Validation - Data Reporting

Subsequent to data reporting, the QA/QC Officer is responsible for comparing analytical worksheets with actual data entered into the final report. The Senior Scientist or designee reviews all data, forms, addenda, notes and reports. All documents, printouts, stripcharts, notes, etc., will reviewed prior to the preparation of the final report. The QA/QC Officer also reviews the quality control report that accompanies the final report.

Reports are reviewed for:

o Completeness results for all parameters requested; detection limits, units, dates, and sample descriptions are complete and correct.

o Consistency - all parameters are reviewed for internal consistency.

10.2.1 Data Reporting Format

Laboratory analytical result summary reports includes appropriate introductory comments, analytical methods summaries, organizational QA/QC outlines and invoices in addition to listing sample analytical results and associated QC data. Sample analytical result reports and associated QC data sheets are prepared in the appropriate standardized form.

10.2.2 Data Archival

All documents produced for a single sample analysis including pertinent quality control information shall be archived at DTC Laboratories to provide a permanent record of these analyses.

11.0 PERFORMANCE AND SYSTEM AUDITS

A system of performance and systems audits will be performed by organizations both external (i.e., IEPA and USEPA) organizations and internal organizations as described below.

11.1 IEPA/USEPA Performance Audits

Performance audits (QA) will be performed by the QA/QC Officer to determine if DTC Laboratories methods are within acceptable control limits, and as an independent assessment of quantitative data.

11.2 Quality Assurance Audits

Performance audits will focus on the adequacy and implementation of procedures used to determine quantitatively the accuracy of total measurement systems and/or components thereof, and the specific validity of the data derived. Systems audits will focus on the effectiveness and efficiency of the total QA/QC program including the quality and productivity of plans, procedures, personnel and their qualifications, facilities, equipment applications, calibration controls, safety programs, data and records systems, and the audit program itself. All audits will be documented. All QA deficiencies discovered during audits will be documented on Corrective Action Request (CAR) forms, logged, addressed to the cognizant manager for resolution. The proposed resolution must be documented by the cognizant manager, approved by the QA/QC Officer, prior to closeout. Implementation of all closeout actions will be verified by the QA/QC Officer.

11.3 DTC Laboratories Audits

DTC Laboratories participates in a number of performance and systems audits, both internal and external, to monitor the capability and performance of the laboratory and its operations.

11.3.1 External Laboratory Certifications. Audits and Approvals

DTC Laboratories maintains an internal system of performance and systems audits to verify the quality of its measurement systems. These audits are conducted on a semi-annual basis as a part of normal laboratory operations. In addition, the laboratory participates in a number of federal, state, and private laboratory certification, audit and/or approval programs in order to demonstrate its analytical capabilities, professionalism, and expertise. Participation in these programs require the laboratory to demonstrate acceptable laboratory performance through satisfactory completion of routine systems and/or performance audits. As a part of its certification by these various federal, state, and private agencies, DTC Laboratories submits to on-site external systems audits. The inspection audits evaluate the adequacy of laboratory personnel, equipment, documentation, and QA/QC. Performance audits require satisfactory blind analyses of unknown intra-laboratory performance evaluation samples. A listing of laboratory certifications, audits, and/or approvals currently maintained by the laboratory follows:

AGENCY	TYPE OF CERTIFICATION
USEPA	Water Supply (WS) Quality Control (for certification)
USEPA	Water Pollution (WP) Quality Control (for certification)
USEPA	Water Supply Quality Control Check (Internal QA/QC)
USEPA	Water Pollution Quality Control
USEPA	Radon Certification
Nucleus	Radon Quality Control Check
nvlap	Program for Asbestos
PAT	Program for Asbestos

11.3.2 Internal Performance Evaluations

DTC Laboratories participates in intra-laboratory performance evaluations administered by the laboratory QC Manager. The QC Manager periodically submits single blind performance evaluation samples from various sources into the laboratory to internally assess and evaluate analytical performance. These single blind performance evaluation standards are generated in-lab by the QC Manager or obtained from various commercial and regulatory sources. These sources and respective analytical parameters are outlined below.

SOURCE	PARAMETERS
USEPA - Environmental Monitoring and Support Laboratory (EMSL)	Trace Metals, Organics, Inorganics, Pollutants
USEPA - Water Quality Survey Performance Standards	Interim Primary Water Quality Trihalomethanes
USEPA - Research Triangle Park	Bulk Asbestos materials
American Industrial Hygiene Association - Performance Audit Testing (PAT)	Metals, Organic Solvents, Asbestos
Analytical Product Group, Inc.	Various Organics and (Commercial) Inorganics

11.3.3 Internal Performance Evaluations

Representative analytical parameters are evaluated on a monthly basis by the QC Manager. Compliance with accuracy and precision standards is monitored, and monthly internal QC control charts are generated.

12.1.4 Routine Maintenance Procedures

DTC Laboratories maintains a routine laboratory equipment maintenance program for all major instrumentation. For GC and GC/MS instruments, selected operators have been trained to perform routine maintenance procedures (i.e. changing oven fans, replacing electronic control boards, changing vacuum pump oil, cleaning quadruple rods, etc.). For the AA and ICP instruments the laboratory maintains a service contract with the manufacturer. For the other instrumentation operators perform routine maintenance (i.e. changing electrodes, changing bulbs, etc.). This program ensures minimal downtime, as well as proper performance. Laboratory instrument service log books are assigned to detail and document the service of all equipment included within this program. A substantial spare parts inventory is also maintained to assure timely repair of instruments. Additional specific preventive maintenance procedures for laboratory instruments are listed below.

12.1.4.1 GC and GC/MS

Utilization of high-quality industrial operating gases combined with on-line installation of molecular sieves and oxygen traps to remove impurities.

o Daily replacement of GC and GC/MS septa (or more frequentlly as needed).

o Daily "bake off" of GC and GC/MS columns and detectors to cleanse system.

o Periodic cleansing and reconditioning of detectors as indicated by operational consistency.

o Constant monitoring of detector response and overall instrument performance through calibration and verification.

12.1.4.1 AA/ICP

o AA lamps shall be warmed up for 15 minutes prior to any analyses.

o Periodic cleaning of windows with alcohol to assure optimal light transmission.

o Daily aspiration of 50 ml of deionized water through the flame assembly or the vapor generation assembly after analyses are complete.

o Thorough wash of the burner assembly and spray chamber in hot water as indicated by instrument response.

o Periodic replacement of pyrolitic graphite furnace tubes as indicated by operational consistency

o Utilization of high quality industrial operating gases.

o Constant monitoring of detector response and instrument performance through calibration and verification.

12.1.4.2 ICP Utilization of high quality operating gases.

o Constant monitoring of detector response and instrument performance through calibration and verification.

o Daily replacement of peristaltic pump tubing.

o Periodic cleaning of nebulizer and spray chamber.

o Daily aspiration of cleaning solution to maintain a clean operating system.

12.4.2 pH and Ion-Selective Electrodes

o Rinse probe with deionized water after every analysis and carefully blot off remaining deionized water prior to next analysis.

o Soaking of probe in a suitable solution when instrument is not operating.

o Periodic replacement of the electrodes as indicated by the consistency, repeatability, and stability of the response.

- Rinse cuvette with deionized water between analyses.

- Periodic cleaning of windows with alcohol to assure optimal light transmission.

- Periodic replacement of lamps as indicated by the consistency, stability, and repeatability of the response.

- Warm up lamp for 10 minutes prior to any analysis.

12.3 Instrument Downtime

Routine maintenance procedures allow the laboratory workload to be scheduled around planned downtime. In the event of unscheduled alternate, certified laboratories. A substantial spare parts inventory is maintained to assure timely repair of instruments and minimize the likelihood of having to send samples out of the laboratory.

13.0 PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

DTC Laboratories use specific procedures to assess the precision and accuracy of monitoring and analytical data. These measures include the validation and internal quality control procedures discussed in Sections 8 and 9.

13.1 Laboratory Analytical Data

Specific procedures for assessing data accuracy and precision include calculation of percent recoveries and relative percent differences for all duplicate spike sample analyses. These calculations are summarized below, and apply to all analytical parameters.

1. Accuracy = % Recovery = $\frac{\text{(Observed Conc.)}}{\text{[Expected Conc.]}} \times 100$
   (R%)
   Relative Percent Difference

2. Precision = R P D = $\frac{|C1-C2|}{1/2(C1+C2)} \times 100$
   (Relative Percent Recovery)
   (Where C1 and C2 are concentrations of duplicate spikes.)

3. Completeness = $\frac{\text{No. of successful analyses x 100}}{\text{No. of requested analyses}}$

Analytical control limits are derived from statistical manipulation of each data category. These limits are outlined below.

	Accuracy	Precision
Upper Control Limit (UCL)	%R + 3S	RPD + 3S
Lower Control Limit (LCL)	%R - 3S	RPD - 3S

Where S is Standard Deviation

Percent recovery determination (%R) are entered into the Laboratory Computer Data Management System. This Laboratory Computer Data Management System will formally record the percent recovery data and calculate the mean, standard deviation and the relative percent difference of each pair, and generate continuous R-S quality control charts of the accuracy and precision for each method commonly used in the laboratory. The quality control charts will provide a continuous indication of method performance by visibly comparing actual quality control recovery data to acceptable USEPA (if available) and/or laboratory method performance criteria.

14.0 CORRECTIVE ACTION

Corrective action procedures to assure that conditions adverse to quality in the laboratories, such as malfunctions, deficiencies, deviations, and errors are promptly investigated, documented, evaluated and corrected.

14.1 Corrective Action

When a significant condition adverse to quality is noted, the cause of the condition will be determined and corrective action will be taken to preclude repetition of the same condition. Condition identification and cause, documented utilized, and corrective action to be taken will be documented by CAR Form (Figure 14-1) and reported to the manager of the area where the adverse condition exists. Implementation of corrective action will be verified by a follow-up action.

All analysts have the responsibility, as part of the normal work duties, to promptly identify and report conditions adverse to quality.

Corrective actions may be initiated, as a minimum, under the following conditions adverse to quality as identified:

1. When predetermined data acceptance standards are not attained.

2. When procedures are performed incorrectly.

3. When equipment or instrumentation is not in proper calibration or is not functioning correctly.

4. When samples and test results are not completely traceable.

5. When QC requirements have not been met.

6. When designated approvals have been circumvented.

7. As a result of issues discovered during system and performance audits.

8. As a result of issues discovered during management assessments of the project.

The Corrective Action Request (CAR) identifies the adverse condition, references any applicable document, and recommends the corrective action to resolve the condition. The issued CAR is directed simultaneously to the QA/QC Manager and to the person in charge of the item or activity for action. This person oversees the corrective action itself, and returns the request response promptly to the QA/QC Officer, affixing his signature and date to the corrective action block after stating the cause of the conditions and corrective action taken. The QA/QC Officer maintains a log for status control of CARs and responses, confirms the adequacy of each corrective action, and verifies its implementation. CARs are maintained for the record.

14.2 Laboratory

The Laboratory will utilize its quality control data to assess the need for corrective action. Frequent review of data permits rapid identification of the source of analytical or sampling error and implementation of corrective action.

14.2.1 Determination of the Need for Corrective Action

Percent recovery determinations from the systematic matrix spike quality control samples must meet the published USEPA or laboratory method performance criteria outlined previously in order to validate and approve a corresponding batch of sample analyses. The method analyses are out of control and therefore unacceptable if:

1. One data point recovery value is outside of published USEPA performance limits or laboratory control limits (CL).

2. Two consecutive percent recovery values are outside of the laboratory warning limits (WL).

3. Seven consecutive percent recovery values are on one side of R line.

4. Percent recovery values increase or decrease

5. Method criteria for surrogates, blanks, and check standards are not met (for SW846 Methods, i.e. 8080, 6010, 7060, 7470, 7471, 7740). Unacceptable values will render the corresponding batch of sample analyses suspect, as will unacceptable results for method blank analyses, until corrective action demonstrates the return of acceptable method performance.

14.2.2 Procedures for Corrective Action

The analyst who reviews and compiles raw data from analyses and associated matrix blank and matrix spike samples must immediately notify the QC Manager of deviation from accepted standards. In addition, the QC Manager reviews all QC data on a monthly basis to monitor the performance of the analytical system. If any values are outside of QC limits, corrective action will be instituted at once.

Corrective action may also be implemented as a result of external performance and systems audits, intra- comparisons, QA project audits conducted by concerned agencies, or other QA/QC activities.

These corrective actions may involve any phase of the analytical or sampling method including: reagent quality, sample extraction, equipment cleaning, instrument calibration and/or performance, calculations, etc. Specific procedures for corrective action are detailed in the Analytical Methods - Quality Control Procedures section of the Laboratory Quality Control Manual.

In the case of out-of-control data for an external project, the Laboratory Manager will promptly notify the QA Officer. The QA Manager will be informed of corrective actions taken and the return of correct method performance.

Figure 14-1

CORRECTIVE ACTION REQUEST (CAR)
CAR Number: _____ Date: _____ To: _____
You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) to prevent it from recurring. Your written response is to be returned to the Quality Assurance Officer by _____ (Date)
Condition Reference Documents Recommended Corrective Actions
Originator _____ Date _____
Response CORRECTIVE ACTION (A) RESOLUTION (B) PREVENTION (C) AFFECTED DOCUMENTS Signature Date
Follow-up: Corrective Action Verifies by_____ DATE_____

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

15.1 Overall Project QA/QC Reports to Management

Periodically, but at least monthly, the QA/QC Officer, shall prepare a summary of the performance of the laboratory performance against this QA/QC Plan. This report shall be addressed to the President of DTC Laboratories, Inc. and a copy sent to the Division Manager and Laboratory Supervisor. Included in the report will be a summary of all corrective action activities, internal and external audit activities and any other quality-related field and laboratory project activities relevant to meeting the quality objectives of the project.

15.2 Laboratory QA Reports

Quality assurance reports will be issued by the laboratory. On a routine basis, the QC Manager and Technical Director will prepare a quality assurance report for laboratory management. This report shall include biweekly assessments of: data accuracy, precision, and completeness; the results of any internal or external systems and performance audits; a description of any significant QA problems and suggested solutions; and the outcome of any corrective actions undertaken.

16.0 REFERENCE DOCUMENTS

The following documents mandate or supply guidance as to the format, general content, and technical specification inclusion in this QA/QC Plan.

1. "Content Requirements for Quality Assurance Project Plan", by Dr. Chen-Wen Tsai, USEPA Region V.

2. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80 (EPA-600/4-83-004), Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, dated February 1983.

3. "Guidance for Preparation of Combined Work/Quality Assurance Project Plans for Environmental Monitoring", OWRS QA-1, USEPA, May 1984.

4. "Test Methods for Evaluating Solid Wastes - Physical/Chemical Methods", SW-846, Office of Solid Waste and Emergency Response, USEPA, Washington, DC 20460, November 1986.

5. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, Environmental Monitoring and Support Laboratory, USEPA, Office of Research and Development, Cincinnati, Ohio 45268, 1979.

6. "Addendum to Handbook for Sampling and Sample Preservation", EPA-600/4-82-029, USEPA, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, 1983.

7. "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, USEPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, 1983.

8. "Characterization of Hazardous Waste Sites - A Methods Manual: Volume II, Available Sampling Methods", Second Edition, EPA-600/4-84-076, USEPA, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada 89114, 1984.

9. "Preparation of Soil Sampling Protocol Techniques and Strategies'', EPA-600/4-83-020, PB83-206979, USEPA, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, Nevada 89114, 1983.

10. "Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater: Selected Analytical Methods Approved and Cited by the USEPA", American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1981.

11. "Proposed Sampling and Analytical Methodologies for Addition to Test Methods for Evaluating Solid Wastes", PB85-103026, USEPA, distributed by National Technical Information Services, U.S. Department of Commerce, Springfield, Virginia 22161

12. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule", 49 CFR 43234, October 26, 1984 and 50 CFR 695, January 4, 1985.

13. "Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual", USEPA Region IV, Environmental Services Division, Athens, Georgia, April 1986.

14. Interim Standard Air Monitoring Guide for Hazardous Waste Sites, U. S. Army Corps of Engineers, June 1984.

15. US-DOT Regulations, 49 CFR 171 through 177.

16. "National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples". U. S. Environmental Protection Agency, Office of Planning and Management, April, 1981.

17. "Handbook for Sampling and Sample Preservation of Water and Wastewater", EPA-600/4-82-029, USEPA, EMSL, Cincinnati, Ohio, September 1982.

18. USEPA Regulations, 40 CFR 136 and 141.

STANDARD OPERATING PROCEDURES
FOR
ASBESTOS DIVISION

DTC Laboratories, Inc.

Department Asbestos - General	Subject Authorization of SOPs
Procedure # A1	Replaces Procedure # of /
Q.A. Managers Signature Date C M Lam 11/1/88	Firm Managers Signature Date Wally K. Safsech 11-1-88

1. Purpose

The purpose of this procedure is to affirm that DTC Labs has authorized the Asbestos Division to prepare and implement Standard Operating Procedures necessary to comply with and insure adherence to good laboratory practices and regulations germaine to the analysis of asbestos.

2. Scope

This procedure discribes the policy regarding issue, use, and interpretation of Quality Assurance SOPs.

3. Procedure

3.1. The Quality Assurance Standard Operating Procedure (SOP) is a document that expresses Management's commitment, as well as Quality Assurances proactives, objectives and controls that are necessary to achieve compliance with good laboratory practices to ensure highest quality results.

3.2. Formal SOPs will be issued using Q.A. Form A - 6.

3.3. The author of SOPs will submit to the Asbestos Q.A. manager for approval.

3.4. The Quality Control Manager will indicate approval be signing signature and date.

3.5. The Firm Manager will indicate concurrence by signing signature and date.

3.6. The SOP will be distributed to the following people:
a. Firm manager
b. Q.A. Manager - Asbestos Division

Department Asbestos - General	Subject Authorization of SOPs
Procedure # A1	Replaces Procedure # of /

Asbestos Analyst

3.7. When the Q.C. Manager and the Firm Manager have signed the SOP, this will indicate that they and all employees are responsible for adhering to the practices described.

4. Responsibilities

4.1. It whall be the responsibility of the Q.A. Manager to assure that there are adequate SOPs to insure compliance with good laboratory practices and to see that the SOPs are porperly implemented and in operation.

4.2. It shall be the responsibility of the Firm Managear to assure that SOPs are properly implemented and carried out by appropriate operational personnel.

4.3. It shall be the responsibility of all concerned in the Asbestos Division to comply with and adhere to quality assurance SOPs.

DAILY ANALYTICAL LABORATORIES

Statement

of

Qualifications

1. LABORATORY EQUIPMENT

Instrument	Model	Quantity	Age	Condition
1.1				
Gas Chromatograph/Mass Spectrometer	HP5970	1	4yrs.	Good
Gas Chromatograph/Mass Spectrometer	HP5995	1	2yr.	Good
Gas Chromatograph/Mass Spectrometer	HP5995	1	1yr.	Good
1.2				
Gas Chromatograph/Dual ECD	PE3920	1	11yrs.	Fair
Gas Chromatograph/FID-NPD	HP5890	1	4yrs.	Good
Gas Chromatograph/ECD	HP5890	1	2yrs.	Good
1.3				
Inductively Coupled Plasma	ARL 3410	1	2yr.	Good
1.4				
GFAA Spectrophotometer	PE2380	1	8yrs.	Fair
GFAA Spectrophotometer	Varian 40	1	1yr.	Good

2. KEY PERSONNEL

Resumes of all key personnel are attached with additional information.

2.1 GC/MS Laboratory Supervisor

Patricia Schultz-Benker

- B.S./1976/Biology
- M.S./1983/Environmental Science
- 4 years Organic analytical experience at Daily Analytical
- 1 year Inorganic analytical experience at Daily Analytical
- 2 years of related chemical experience with other firms

2.2 GC/MS Operators

James J. Lamkin

- B.A./1979/Biology
- M.S./1984/Environmental Studies
- 3 years experience at Daily Analytical
- 3 years of related chemical experience with other firms

Lori L. Stenzel

- B.S./1983/Biology
- 2 years experience at Daily Analytical

Larry A. Drake

- H.S./1983
- 4 years experience at Daily Analytical

Kay Ingels

- B.A./1975/Chemistry
- 3 years experience at Daily Analytical
- 9 years of related chemical experience with other firms

2.3 Pesticide Residue Analysis

Doug J. Beisinger

- A.S./1987/Chemistry
- 2 years experience at Daily Analytical

Stephanie L. Lannert

- B.S./1989/Microbiology
- 1 year experience at Daily Analytical

2.4 Spectroscopy (Trace Metals) Laboratory Supervisor

Dave J. Cirilli

- B.S./1986/Pharmacy
- 2 years experience at Daily Analytical
- 5 years of related chemical experience with other firms

2.5 ICP Operator

H. Keith Cressman

- B.A./1948/Chemistry
- M.A./1954/Agronomy
- Ph.D./1961/Soil Science
- 5 years experience at Daily Analytical
- 25 years of related chemical experience with other firms

2.6 Graphite Furnace AA Operators

Shaun A. Riedell

- B.A./1984/Communication
- 2 years experience at Daily Analytical

Tom McMillan

- B.S./1975/Chemistry
- 2 years experience at Daily Analytical

2.7 Extraction/Concentration (Organic Analysis)

Doug A. Hafley

- B.S./1989/Chemistry
- 3 years experience at Daily Analytical

Curtis W. Means

- B.S./1986/Chemistry
- 3 years of related chemical experience with other firms

2.11 Metals Digestion

Tom McMillan

- B.S/1975/Chemistry
- 2 years experience at Daily Analytical

3. LAYOUT OF FACILITIES

A blueprint of our 8000 sq.ft. facility has been provided. Various areas have been numerically labelled on the blueprint.

3.1 Sample Receipt Area

The sample receipt area is at the main entrance of the building. There are 33 sq.ft. of bench top available for receipt and logging of samples. The bench top is constructed of durable formica which can be easily cleaned between sample batches. The receipt area is adjacent to the semivolatile and inorganic sample walk-in cooler. The receipt area itself has adequate storage space for precleaned sample bottles and shipping containers.

3.2 Storage Area

Four separate storage areas are maintained. The walk-in cooler for semivolatiles and inorganics (# 20 on blueprint) has an internal volume of 665 cubic ft. The volatile samples are stored in a separate 366 cubic ft. walk-in cooler (# 19 on blueprint). Both of these coolers have shelving constructed such that samples can be stored chronologically by D/A sample identification. Both of these coolers are locked when qualified personnel are not present.

Two refrigerator/freezers are maintained in the Organics Section. One contains sample extracts and the other is used to house calibration standards.

3.3 Ventilation System

The laboratory is divided into six (6) heating/air conditioning zones. These zones are:

a) Northwest Wet Chemistry Annex and Organic Extraction Lab
b) Trace Metals Lab and Sample Receiving Area
c) Gas Chromatography Lab and Bottle Storage
d) Wet Chemistry Laboratory
e) Office Areas on the east side
f) GC/MS Laboratory

The zones minimize the possibility of airborne contamination between laboratory sections.

3.4 Benchs and Exhaust Hoods

Benches are labelled in the blueprint. All benches are either chemical resistant stone or epoxy clad wood. Ventilation hoods are present in the Trace Metals area (# 10 on blueprint) and Organic Sample Preparation Lab (# 18 on blueprint). A unique capture hood for vapors, fumes, and heat covers an entire bench in the main Wet Chemistry Laboratory (# 17 on blueprint), while a second similar capture hood is found in the Wet Chemistry Annex (northwest corner of building). This second capture hood is dedicated to work where chlorinated solvents are used, such as oil and grease and phenol extractions. In addition, a venting system for a GC and GC/MS exhaust transports any gases to the outside of the building.

3.5 Deionized Water

Laboratory pure water is generated from a Milli-Q System,
and routinely generates 18megohm water.

Commercially obtained mixed bed resin tanks feed the Milli-Q System after passing the 0.22 micron filter. The Milli-Q System contains two ion exchange cartridges, three organic removal cartridges, and a final 0.22 micron filter.

Routine QC checks of the water are as follows:

daily -
- Resistance minimum 10 Megohms

monthly -
- pH: between pH 5.5 and 7.5
- Standard Plate Count: <1,000 #/ml
- Chlorine Residual: <0.1 mg/l
- Total Organic Carbon: <1.0 mg/l

annually -
- Suitability: non-toxic and non-stimulating
- Trace Metals: <0.05 mg/l

Maintenance Record

Documentation is kept to track:

1) Supply tank replacement
2) Daily consumption in gallons
3) Resin beds and organic filter replacement
4) 0.22 micron filter replacement

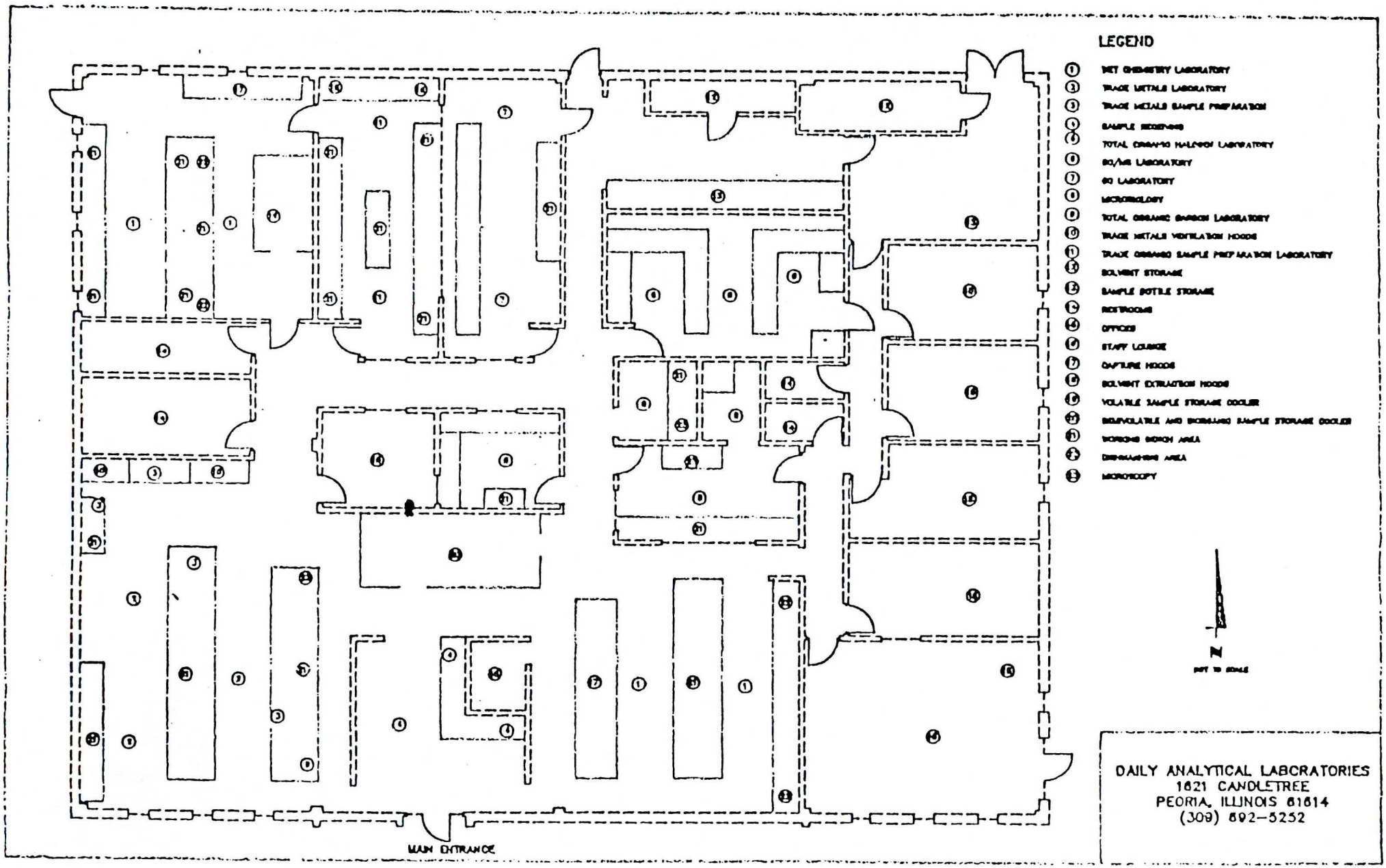
3.6 Analytical Balances

Five analytical balances are utilized in the laboratory. One high sensitivity balance is present in the main Wet Chemistry Laboratory. It is draft shielded and is placed on a vibration insulating block table. A second high sensitivity balance is present in the Gas Chromatography Laboratory. It rests on a vibration damping stone base. It is also hooded and vented to both minimize drafts and to control any vapors emitted during weighing of organic standards. Three toploading balances are maintained in the laboratory for sample weighing. All three are located such as to minimize the affects of drafts, temperature change, and vibrations on weighings.

4. LABORATORY ANALYTICAL EXPERIENCE

In the past seventeen (17) years, Daily Analytical has performed a tremendous variety of analyses. Analyses have included traditional wastewater tests, point source monitoring for air emissions, groundwater monitoring, hazardous waste determinations, forensic investigations, qualitative identification of unknown materials and determinations of organic materials, sometimes at sub-part per billion levels. We have handled unique and custom analyses with ingenuity and thoroughness. We have also handled large sampling programs involving hundreds of samples.

Our projects regardless of size receive the same careful consideration.


LEGEND
MAIN ENTRANCE
N
DAILY ANALYTICAL LABORATORIES
1621 CANDLETREE
PEORIA, ILLINOIS 61614
(309) 692-5252

QUALITY ASSURANCE PLAN

Prepared March 5, 1990

by

DAILY ANALYTICAL LABORATORIES

Kurt C. Stepping
Chief Chemist

Sarah E. Vesonder
Quality Assurance Coordinator

TABLE OF CONTENTS

I. CHART OF ORGANIZATION AND INDIVIDUAL RESPONSIBILITIES

Chief Chemist

Kurt C. Stepping, is responsible for co- managing the laboratories with Doug Bischoff. He also functions as a project chemist. He is responsible for evaluating all data produced from the Inorganic and Trace Metal Laboratories.

Senior Project Manager

Douglas R. Bischoff, is responsible for co-managing the laboratories with Kurt Stepping. He is also responsible for coordinating several major analytical projects as well as most of the business aspects of the laboratories.

Senior Staff Chemist - Inorganic Chemistry

Judith A. Marshall, has primary supervisory responsibility of the Inorganic Chemistry Laboratory. She also serves as a project chemist.

Senior Staff Chemist - Trace Metals

Dave J. Cirilli, has primary supervisory responsibility for the Trace Metals Laboratory. He also serves as a project chemist.

Senior Staff Chemist - Organic Chemistry

Patricia Shultz-Benker, has primary supervisory responsibility for the Organic Chemistry Laboratory. She is responsible for all data generated from that laboratory.

Sample Team Leader

Scott A. Grubisich, coordinates the sampling plans for all the clients. He oversees the field crew in preparation and execution of the projects involving groundwater monitoring, wastewater monitoring, and soil sample collection. He also reviews the analytical reports for several of those same monitoring programs, particularly groundwater monitoring.

Project Chemist - Bacteriology and Asbestos

Stephen E. Julien, has primary responsibility for the Bacteriology and Asbestos Section within the laboratory. Mr. Julien is responsible for the technical evaluation of performance within these sections.

Quality Assurance Coordinator

Frank J. Calovini, has responsibility for reviewing analytical data produced in all sections of the laboratory with respect to Quality Control practices. He audits the operations of the separate laboratories and provides recomendations to the supervisors and laboratory director.

Assistant Project Chemist

Dave R. Calhoun, has primary responsibility in overseeing a number of analytical projects. He insures that these projects run smoothly and communicates any problems to both management and the client.

Analysts

Analysts have primary responsibility for the collection of data. It is their responsibility to collect valid data, to follow the defined analytical protocol, and make routine assessments of the system Precision and Accuracy.

Supervisors

Supervisors have primary responsibility for a specific area of work. It is their responsibility to oversee a group of employees, review analytical data from their section, and act as a Project Chemist for their section.

Project Chemists

Project Chemists have responsibility for organizing and monitoring analytical projects. It is their responsibility to act as a liaison with the client and communicate any pertinent information.

PERSONNEL	DEGREE	MAJOR	FUNCTION	AREA OF RESPONSIBILITY
Kurt C. Stepping	B.S.	Chemistry	Chief Chemist	Project Administration
Stephen E. Julien	B.S.	Microbio.	Environmental Scientist	Microbiology/ Microscopy
Douglas R. Bischoff	B.S.- M.B.A.	Chemistry -Management	Operations Director	Project Administration
H. Keith Cressman	Ph.D.	Chemistry	Senior Staff Chemist	ICP Trace Metals
Patty Schultz-Benker	M.N.S.	Environ. Science	Supervisor	Trace Organics
Andy J. Groeper	B.S.	Marketing	Marketing Director	Marketing
James J. Lamkin	M.S.	Environ. Science	Mass Spectroscopist	Trace Organics
Doug J. Burk	B.S.	Biology	Staff Chemist/ Field Sampling	Inorganics/ Field Sampling
Mark A. Williams	B.S.	Geology	Staff Chemist Field Sampling	Inorganics/ Field Sampling
Doug J. Biesinger	A.S.	Chemistry	Analyst	Trace Organics
Scott A. Grubisich	B.A.	Chemistry	Field Project Chemist	Field Projects Sampling
Judith A. Marshall	M.S.	Nat. Science Biochemistry	Supervisor	Inorganics
Amy J. Tanner	A.S.	Dental Assistant	Analyst	Asbestos
Kay Ingels	B.A.	Chemistry	Senior Organics Chemist	Trace Organics
Sharon J. Hammer	A.S.	Psychology	Secretarial	Clerical
Doug A. Hafley	B.S.	Chemistry	Staff Chemist	Trace Organics
Dorothy J. Dunn	H.S.		Office Manager	Admin./Clerical Billing
Donna A. Wenner	B.S.	Chemistry	Staff Chemist	Inorganics
Todd L. Reed	H.S.		Glassware/ Maintenance	Bottle Q.C.

PERSONNEL	DEGREE	MAJOR	FUNCTION	AREA OF RESPONSIBILITY
Sarah E. Vesonder	B.S.	Chemistry	QA Coordinator	QA/QC
Lori L. Stenzel	B.S.	Biology	Mass Spectroscopist	Trace Organics
Dave J. Cirilli	B.S.	Pharmacy	Supervisor	Trace Metals
Elaine A. Kaufman	B.S.	Biology	Staff Chemist	Inorganics
Ed L. Hicks	B.S.	Biology	Staff Chemist	Inorganics
Dave J. Hampson	B.A.- B.S.-	Biology Pharmacy	Sample Control Specialist	Sample Reception
Walter Hogan	H.S.		Glassware	Glassware Preparation
Herman E. Daves	H.S.		Glassware/ Maintenance	Bottle Q.C.
Lori J. Horstman	A.S.	Applied Science	Bacteriologist	Asbestos/ Microbiology
Dave R. Calhoun	B.S.	Agriculture Animal Sc.	Project Manager	Project Management
Jeff M. Loewe	B.S.	Chemistry	Staff Chemist	Inorganics
James T. McMillan	B.S.	Chemistry	Staff Chemist	Trace Metals
Shaun A. Riedell	B.S.	Communication	Analyst	Trace Metals
Larry A. Drake	H.S.		Mass Spectroscopist	Trace Organics
Stephanie L. Lannert	B.S.	Chemistry	Staff Chemist	Trace Organics
Janice A. Behrends	H.S.		Purchasing Agent	Purchasing
Brent D. Litwiller	B.S.	Chemistry	Staff Chemist	Inorganics
Curtis W. Means	B.S.	Chemistry	Staff Chemist	Trace Organics
Kevin D. Clendenny	H.S.		Bottle Washing/ Maintenance	Bottle Q.C.

II. QA OBJECTIVES IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

A. General Approach

Quality Control refers to those practices performed at the analytical level to measure the validity of the data generated. Quality Control is discussed in other sections, principally in section VIII, Internal Quality Control Checks.

Quality Assurance refers to those practices performed by management to measure the level of Quality Control. QA/QC practices do not, per se, insure the quality of data.

The purpose of this Quality Assurance Plan is to provide a uniform basis for bottle washing, sample collection, chain of custody, analytical procedures, data validation, data reduction, reporting, and archiving. Properly understood, Quality Assurance begins before sample bottles are sent to the site and continues after the data is archived.

B. Acceptance Criteria

Two standard measures of quality are Precision and Accuracy. Precision refers to the "reproducibility" of a procedure and is measured by Relative Percent Difference (RPD) of duplicate sample analyses. Accuracy refers to the "closeness" of a measurement to a defined standard and is measured by Percent Recovery (%R) of sample spike analyses. RPD an %R are defined in section XI, Specific Routine Procedures used to Assess Data Precision, Accuracy and Completeness.

C. Acceptance Limits

The following limits are established for goals in measurement of Precision and Accuracy.

Parameter	Precision - RPD	Accuracy - %R
Trace Metals	+/- 20%	100% +/- 25%
Cyanide	+/- 20%	100% +/- 25%
Nitrate	+/- 20%	100% +/- 25%
Phosphorous	+/- 20%	100% +/- 25%
Total Organic Carbon	+/- 20%	100% +/- 25%

Parameter	Precision - RPD	Accuracy - %R
Total Organic Halogen	+/- 20%	100% +/- 25%
VOA's	(1)	(1)
Base-Neutrals	(1)	(1)
Acids	(1)	(1)
Pesticides/PCB's	(1)	(1)

(1) Matrix Spike/Matrix Spike Duplicates are performed on all Organic analyses in a frequencey of 5% of all samples processed. RPD's and %R's are calculated and reported. Acceptance criteria for these measurements are advisory only and have no bearing on sample reanalysis. Surrogate spiking is performed with each sample and those %R's used as criteria for data acceptability.

III.

SAMPLE CONTAINERS, SAMPLE PRESERVATION, REQUIRED VOLUME, AND HOLDING TIME

Parameter	Size and Type of Container	Preservative1	Volume Needed	Holding Time2
Ignitability	1 quart wide mouth	refrigerated 4 Centigrade	100ml	25 days
Corrosivity	1/2 gallon plastic	refrigerated 4 Centigrade	100ml	6 hours
EP-Toxicity	1 quart wide mouth	refrigerated 4 Centigrade	1000ml	25 days
Aqueous Metals Except Cr+6	1 liter plastic	5 ml conc. HNO3 acid	1000ml	38 days
Cr+6	1/2 gallon plastic	refrigerated 4 Centigrade	100ml	24 hours
VOA's or VOC's	40 ml vials (2 per sample)	3 drops 0.5N Na2S2O3	2-40ml	14 days
Semi-Volatiles B/N/A's	1/2gallon amb. (2 per sample)	refrigerated 4 Centigrade	2000ml	14 days3
Cyanide	1 liter plastic	5 ml of 6N NaOH	500ml	14 days
Total Organic Halogen(dup)	250 ml amber (2 per sample)	0.5 ml H2SO4/ 250 ml	500ml	25 days
Total Organic Halogen(quad.)	500 ml amber (2 per sample)	1 ml H2SO4/ 500 ml	1000ml	25 days
Total Organic Carbon(dup)	40 ml amb.vial (2 per sample)	0.25ml H2SO4 per vial	2-40ml	28 days
Total Organic Carbon(quad.)	250 ml amber (2 per sample)	refrigerated 1.0ml H2SO4	500ml	28 days
[illegible]rimetric [illegible]fide	1 liter plastic	2ml Zn Acetate 5ml of 6N NaOH	1000ml	7 days
pH	1/2 gallon plastic	refrigerated 4 Centigrade	100ml	Analyze Promptly
Pesticides PCB's	1/2gallon amb. (2 per sample)	refrigerated 4 Centigrade	2000ml	14 days3
Herbicides	1/2gallon amb. (2 per sample)	refrigerated 4 Centigrade	1000ml	14 days3
Phenol Wastewater	250 ml amber	0.5ml H2SO4	250ml	28 days
Phenol Groundwater	1 liter amber	2.0ml H2SO4	1000ml	Analyze Promptly
Grease and Oil	1 Liter Glass	5.0 mL H2SO4	1000ml	28 days
COD, TKN, NH3,NO3 Total Phosphorous	500 ml plastic	2 ml H2SO4	500ml	25 days
Total Organic Carbon(Hi Sens.)	250 ml amber	refrigerated	250ml	4 days
Asbestos	Two 100 mL plastic bags	none	25 g.	indefinite

1 Preservatives used only if the sample is aqueous.

2 Sample Preservation and Holding Times not specifically identified will be done in accordance with the Federal Register, Oct. 26, 1984, p. 43260, Table II.

3 Extraction to be accomplished within 14 days. Extract may be stored for 40 days at 4C.

IV. CHAIN OF CUSTODY

A). Sample Logistics - The client will be provided with a list of key personnel and their home phone numbers. At least one individual from this list will be available at all times. Such a listing is included as Appendix A.

B) Sampling Team Custody Provisions -

1. The Sample Team Leader and his crew will be provided with the properly prepared sample bottles for the sample points to be collected. A chain of custody form will be provided with each sample cooler. A specific cooler number will appear on the chain of custody form identifying the number of containers present per sample point.

2. Sample bottles will be coded with a date of Quality Control analysis on the parameter/preservation label. This date identification will serve as means for retracing sample bottle quality back to initial Quality Control analysis as performed in "SAMPLE BOTTLE PREPARATION AND QUALITY CONTROL".

3. The individual sample coolers will be checked by a Quality Assurance Section member of Daily Analytical Laboratories to assure that the cooler and its contents are complete. He will then sign the chain of custody form along with the date and time of the inspection. He will also identify the custody form with the number specified on the cooler. After the custody form and cooler are inspected, the custody will be transferred to the sampling team or sample team leader.

4. The Sample Team leader will then retain custody of the coolers and provide information as specified on the chain of custody form.

5. The sample coolers and the samples themselves will be in the custody of the Sample Team Leader from the laboratory through sample collection and remain so until delivery is made back to the laboratory. If other transport or delivery is necessary other than by field personnel, time and date and signatures of the transfer of custody must be indicated on the chain of custody form. Couriers such as, UPS, Emery, etc. will not be required to sign for custody. In the event that transport must be made by courier service, the sample team leader will seal the cooler(s) with evidence tape prior to release to the courier. The chain of custody will be sealed inside the cooler accompanying the samples.

6. The Sample Team Leader will be responsible for logging the samples into the laboratory upon delivery, provided that the Sample Team Leader is a member of the laboratory staff.

7. Trip Blanks will be provided by our laboratory at the frequency specified in the Sample Analysis Plan.

C). Chain of Custody - A sample is in someone's "custody" if:

1. It is in their actual physical possession.
2. It is in their view, after being in their physical possession.
3. It was in their physical possession and is secured so that no one can tamper with it.
4. It is kept in a secured area restricted to authorized personnel only.

D). Laboratory Custody Procedures - The laboratory responsibility for sample security and integrity begins with the delivery of the samples to the lab. Samples are stored in two (2) walk-in coolers. Access to the general laboratory is limited to Clients and Staff. Non-employees are prohibited from the cooler, unless accompanied by an Employee. Coolers are locked when the lab is unoccupied.

E). Log - In Procedures

1). Sample collectors bring samples to the reception area.

2). The collector opens each cooler separately and verifies that the cooler custody seal number corresponds to the number indicated on the enclosed custody form.

3). Lab personnel check coding on sample containers to ensure that samples are properly coordinated with the custody forms.

4). Lab numbers are assigned to the report form and these numbers are plainly marked on the appropriate sample containers.

5). Clients or representatives of the client delivering the samples are asked to wait until sample numbering is performed and a complete check is made on the samples and respective chain of custody forms.

6). Lab personnel sign for each sample on each custody form and at this time note on the report any deviations from sample preservation procedures.

7). Sample information is logged into a master log book. A unique job order number is assigned to each sample. Sample identification and designation, date and time of collection, date and time of receipt, and chain of custody information are recorded onto Laboratory Work Sheets and the laboratory computer system.

8). Sample distribution in the lab is carried out by the receiving section to ensure that samples are sent to proper areas for analyses.

9). When containers, samples, or extracts are transferred from one laboratory to another, the appropriate chain of custody sign out/in protocol must be used. Samples or extracts should be shipped in sealed containers, following the appropriate DOT regulations where necessary.

10). Samples shall be retained in cold storage for a maximum of two weeks after completion of the final report. After this time period the samples will be either shipped back to the client or properly discarded. The client will be responsible in notifying the laboratory within the two week period if they wish to have cold sample storage extended beyond this time.

11). All Asbestos samples are returned to the client after their report has been found to be satisfactory.

F). Sample Custodian - Asbestos

Amy J. Tanner, has primary responsibility for the custody of all samples received for asbestos analysis. She documents the arrival and eventual disposal or return of all samples received for analysis. She is responsible for security and maintenance of all pertinent records associated with asbestos samples.

All Asbestos testing complaints are forwarded to either Steve Julien or Susan Naschert. If warrented the disputed sample will be repeated by two analysts and any discrepancies noted. If the analytical result is still in question the sample will be sent to a qualified neutral laboratory.

Sample acceptance/rejection criteria - The sample custodian has the responsibility of rejecting any sample which does not meet the laboratories criteria for acceptance.

Rejection criteria : Broken evidence tape
Improper containers (paper bags, broken plastic bags, etc...)
Excess of sample

V. A. CALIBRATION PROCEDURES AND FREQUENCY - INORGANICS

Definition of Terms

1) Preparation Blank - an aliquot of lab pure water subjected to the entire analytical scheme (in methodologies requiring sample digestion, distillation, or extraction) which monitors for contamination attributable to preparation. Performed with each analytical batch or twenty (20) samples, but not less than the latter frequency.

2) Laboratory Control - an aliquot of lab pure water spiked with a known amount of analyte that is subjected to the entire analytical scheme (in methodologies requiring sample digestion, distillation, or extraction) which assures the validity of the method being used. The frequency is a minimum of once every fifteen (15) samples processed. Control limits are set at 80-120% recovery of the analyte.

3) Check Standard - a standard which is used to verify a Calibration Curve. This standard is always from a different stock source than the standard which is used to prepare the working standards. Its known value "checks" the integrity of the standard used for calibration.

4) Sample Duplicates - two independently processed aliquots of the same sample.

Sample duplicates are performed at a frequency of 10% of the samples processed. Control limits for duplicate analyses are set by Relative Percent Difference of the replicate values. For samples above five (5) times the detection limit, the criteria at +/-20%. For replicates with results both below five (5) times the detection limit the acceptance criteria are set at replicate values of +/- 1 detection limit value. If one value is below five times the detection limit and the other is above five times the detection limit, then the criteria of +/- 1 detection limit is used. If either or both replicate values are below the detection limit, the RPD is reported as non-calculable (N.C.).

Limits exceeded in the initial duplicate analysis will require the duplicates be performed a second time. A second duplicate analysis out of compliance with the limits will require that the final result be flagged with a *.

5) Sample Spikes - an aliquot of the sample treated with the analyte of interest to measure recovery.

Sample spikes are performed at a frequency of 5% of all samples processed. Acceptance limits are set at 75-125% recovery of the spike. These acceptance limits are not applicable when the concentration of the analyte in the sample exceeds the concentration of the spike by greater than four times. If 75-125% recovery limits are exceeded and the analyte concentration (> 4 times the spike) of the sample is not an issue then the sample is reanalyzed once (beginning with digestion or distillation). A second recovery exceeding the limits will result in flagging the result with a "R" and supplying the client with the data from the analysis which contains the closest % recovery to the acceptance limits.

Calibration for Colorimetric Procedures

Individual Analyst Demonstration

A) An Initial Calibration Curve of a Calibration Blank and five (5) working standards covering the analytical range of the spectrophotometer.

- correlation coefficient must be greater than 0.995

B) An Initial Verification (Check Standard) performed in parallel with the Initial Calibration Curve.

- % Recovery of the Check Standard at 90-110% or within acceptance limits published with the source.

Subsequent Calibration

A) Continuing Calibration Curve composed of a Calibration Blank and two working standards(ideally at mid and upper range points of the spectrophotometer).

- correlation coefficient greater than 0.995
- slope of points within +/- 10% of Initial Calibration Curve slope

B) Continuing Verification with a check standard not required.

C) Preparation Blank to be analyzed with every 20 samples which are processed through a digestion or distillation.

D) Lab Control Sample to be analyzed with every 15 samples which are processed through a digestion or distillation.

E) A sample duplicate measured for every 10 samples processed.

F) A sample spike measured for every 20 samples processed.

Calibration for Potentiometric Analyses

Individual Analyst Demonstration

A) An Initial Calibration Curve of a Calibration Blank and five (5) working standards covering the analytical range of the meter.

- correlation coefficient must be greater than 0.995 if using millivolt or relative millivolt readings.
- 90-110% recovery of standards outside of the ones used for meter calibration. Correlation coefficient criterion not applicable.
- slope of meter must be within manufacturer's specified limits.

For pH Calibration, use two buffers to calibrate the instrument.

B) An Initial Verification (Check Standard) performed in parallel with the Initial Calibration Curve.

- % Recovery of the Check Standard at 90-110% or within acceptance limits published with the source.

For pH use a third buffer for verification. Acceptance +/- 0.1 unit.

Subsequent Calibration

A) Continuing Calibration consisting of two working standards.

- correlation coefficient must be greater than 0.995 if using millivolt or relative millivolt readings.
- 90-110% recovery for standards other than the ones used for meter calibration. Correlation coefficient criterion not applicable.
- slope of meter must be within manufacturer's specified limits.

For pH Calibration, use two buffers to calibrate the instrument.

B) Continuing Verification consisting of a Check Standard.

- % Recovery of the Check Standard at 90-110% or within acceptance limits published with the source.

For pH use a third buffer for verification. Acceptance +/- 0.1 unit.

C) Analyze Preparation Blank if samples were distilled or digested. Minimum of one for each 20 samples having been distilled or digested. Detection limits for the test are acceptance limits.

For samples processed without preliminary preparation, analyze a reagent blank after each set of 20 samples.

D) Analyze Lab Control Sample if samples were distilled or digested. Minimum of one for each 15 samples having been distilled or digested. Acceptance limits are 80-120% recovery.

For samples processed without preliminary preparation, analyze a working standard or verification standard after each set of 15 samples. Acceptance limits are 90-110% recovery or limits published with the check standard.

Calibration for Titrimetric Analyses

Analyst Demonstration

A) Initial Calibration or Standardization of Titrant.

Standardize titrant against three (3) aliquots of primary standard.

- three consecutive Normalities derived must be within +/- 0.0003 N

B) Initial Verification determined by analysis of a Check Standard.

- acceptance limits are 90-110% recovery or within limits published with the source

Subsequent Analyses

A) Standardize titrant against one (1) aliquot of primary standard.

- normality derived must be within +/- 0.0003 N of Initial Normality or restandardization is required. Analyst may titrate a second aliquot and analyze a check standard and proceed if limits for both are acceptable.

B) Analyze a reagent blank with each batch of 20 samples processed or at a minimum of once per batch whichever is more frequent. Acceptance limits are detection limit based on maximum volume of samples used for titration. Usually, 50 milliliters.

C) Perform a duplicate sample for each 10 samples processed.

D) Sample spikes (by dilution or addition) should be performed at the discretion of the analyst and/or supervisor of the area.

Calibration for Gravimetric Analyses

A) Balance must be calibrated daily. Primary Balance used for gravimetric analyses is the Mettler AE 160. An internal weight calibrates the balance at 100.0000 grams.

B) Balance must be rezeroed prior to each weighing.

C) The following gravimetric procedures indicate the use of either Preparation Blanks or Check Standards routinely.

Parameter	P. Blanks	Check Standards
Total Dissolved Solids	Yes	No *
Sulfate	Yes	Yes
Suspended Solids	Yes	No *
Grease & Oil	Yes	No *

* Check Standards run approximately 1 in 50.

Preparation Blanks must be performed with each batch of samples. Acceptance limits are determined by the detection limits of the test based on the maximum sample volume used.

Check Standards must be performed quarterly for each of the tests. Acceptance limits are those provided by the source of the reference standards used.

D) Sample Duplicates are required at a minimum of one for each ten samples analyzed. This requirement is not observed for aqueous samples tested for Grease & Oil, unless adequate sample is provided.

E) Sample Spikes are not required for the gravimetric procedures. Such spiking is open to the discretion of the analyst and/or supervisor of the area.

V. B. CALIBRATION PROCEDURES AND FREQUENCY -TRACE METALS

1) Initial Calibration

This calibration is performed for each metal in each analytical series. A sensitivity check is performed for the analyte to check optimization of the instrument. Unless this criteria is met, further calibration may not proceed.

Upon successful determination of the sensitivity check, the analyst will demonstrate a four (4) point calibration curve for the analyte. A reagent blank and three (3) working standards covering the test range are aspirated. All four points and their respective responses are plotted or subjected to linear regression and the slope and correlation coefficient of the line are calculated. The correlation coefficient must be greater than 0.995 or the calibration curve repeated until an acceptable coefficient is achieved.

2) Initial Calibration Verification (check standard)

This is performed after the calibration curve has been established. An independent standard (check standard) is analyzed and its recovery quantified. The (check)standard must fall within the calibration range. Limits of acceptance are measured in terms of % recovery. All metals with the exceptions of Mercury and Tin must be recovered at 90-110%. Mercury and Tin recovery limits are 80-120%.

3) Calibration Blank

A calibration blank or reagent blank is measured and quantified. The value obtained may not exceed the window of +/- the detection limit of the parameter analyzed. Analysis must be terminated and the problem corrected. Recalibration must be performed.

4) Preparation Blank

A preparation blank is set with each batch of 20 samples through the digestion process. The analysis of the preparation blank should follow the calibration blank in the analytical sequence.

5) Laboratory Control Sample

The lab control sample is set with each batch of 20 samples through the digestion process. Acceptance for the sample on all metals is 80-120% recovery. This sample is to be analyzed only once with the associated samples. Mercury does not need to have a laboratory control sample performed.

6) Sample Analysis

Ten samples are analyzed or if less than ten samples are analyzed the analyst will proceed to steps 7 and 8.

After each analytical set of ten samples the Calibration Blank and Check Standard are reanalyzed and quantified. If criteria are acceptable proceed to the next ten samples. If a batch contains more than twenty samples, then a second Preparation Blank and Lab Control sample are to be included for each set of twenty samples. Those must be prepared with each batch of twenty samples.

7) Calibration Blank

A calibration blank is reanalyzed. Acceptance windows for the calibration blank is set at +/- the required detection limit for the analyte.

8) Continuing Verification (check standard)

The check standard is reanalyzed. Acceptance limits are the same as in Initial Verification.

MATRIX DEPENDENT QUALITY CONTROL

Duplicate sample analysis will be done on 10% of all samples of each matrix within a batch processed (10 waters and 10 solids). Acceptance criteria are measured in terms of Relative Percent Difference (RPD) and those limits are set at +/- 20% for samples with duplicate values above five times the detection limit. If one or both of the sample duplicate values are less than five times the detection limit (yet above the detection limit) the acceptance criteria will be +/- 1 detection limit. If one or both of the values are below the detection limit, the RPD is non-calculable and should be recorded as N.C.

Sample spike analysis will be performed on 5% of the samples processed of each matrix (20 waters and 20 solids). Acceptance criteria is measured by %recovery of the analyte and is set at 75-125%. This criteria will not be in effect when samples contain a four times or greater analyte concentration than the concentration used to spike the sample.

V. C. CALIBRATION PROCEDURES AND FREQUENCY - ORGANICS

GC/MS VOLATILES

1). Tuning - checked each 12 hours, all criteria within limits.

BFB (*) Tuning Verification Limits

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( )1
174	Greater than 50.0% of mass 95	
175	5.0 -9.0% of mass 174	( )1
176	Greater than 95.0%, but less than 101.0% of mass 174	( )1
177	5.0 - 9.0% of mass 176	( )2

1-Value is % mass 174 2-Value is % mass 176

* p-bromo-fluorobenzene

2). Calibration shall be done by internal standard methods.

3). Continuing Calibration - each day.
All SPCCs meet criteria RF > 0.3 (0.25 for Bromoform.)
All CCCs meet criteria %D < 25% compared to initial calibration.

4). Blank -
VOAs - at least one per day, per matrix.
Additional blanks may be performed among samples to check carryover contamination from heavily contaminated samples.

5). Surrogates - added to all samples.
VOAs - all must be in recovery limits. If not, the analysis is repeated once, and best result reported.

6). Matrix Spike/ Duplicates
One set of spikes per matrix, per 20 samples.
The MS/MSD Summary form contains recommended Recovery Limits and Relative Percent Differences. These acceptance limits are advisory, only. No corrective action is required.

GC/MS SEMI-VOLATILES

1). Tuning - checked each 12 hours, all criteria within limits.

DFTPP (**) Tuning Verification Limits

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	
68	Less than 2.0% of mass 69	( )1
69	Mass of 69 relative abundance	
70	Less than 2.0% of mass 69	( )1
127	40.0 - 60.0% of mass 198	
197	Less than 1.0% of mass 198	
198	Base peak, 100% relative abundance	
199	5.0 - 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	
365	Greater than 1.00% of mass 198	
441	Present, but less than mass of 443	
442	Greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( )2

1-Value is % mass 69 2-Value is % mass 442

** decafluoro-triphenylphosphine

2). Calibration shall be done by internal standard methods.

3). Continuing Calibration - each day.
All SPCCs meet criteria RF > 0.05.
All CCCs meet criteria %D < 25% compared to initial calibration.

4). Blank - one per matrix per day of extraction or one per set of 20 samples, whichever is more frequent.

5). Surrogates - added to all samples.
- A maximum of one surrogate recovery is allowed to fall outside of recovery limits per analysis. If not, the sample analysis is repeated once, and the best result reported.

6). Matrix Spike/ Duplicates
One set of spikes per matrix, per 20 samples.
The MS/MSD Summary form contains recommended Recovery Limits and Relative Percent Differences. These acceptance limits are advisory, only. No corrective action is required.

GC-ECD

1). DDT retention time $\geq$ 12 minutes.

2). Linearity check each 72 hour sequence.
Acceptance based on < 10% RSD.
If only DDT outside limit, then 3-point calibration curve for DDT, DDD, and DDE.

3). Single point calibration factors.
At least one standard re-analyzed per every ten samples.
At least one aroclor standard (usually 1254).
Additional aroclor standards and repeat 1254 if PCBs present in sample, so that response factor is taken from aroclor present in sample.

4). Procedural blank
One blank per day, per matrix or one per set of 20 samples, whichever is more frequent.

5). Surrogate added to each sample for pesticide analysis.
% Recovery is monitored, but not a criterion for repeat analysis.
Retention time of surrogate is also monitored. If outside the acceptance window, sample is re-analyzed.
Best result reported.

6). Matrix Spike/ Duplicates
One set of spikes per matrix, per 20 samples.
The MS/MSD Summary form contains recommended Recovery Limits and Relative Percent Differences. These acceptance limits are advisory, only. No corrective action is required.

V. D. CALIBRATION PROCEDURES AND FREQUENCY - ASBESTOS

1). Microscope Calibration

This calibration is performed each time prior to acquisition of analytical data.

A. Focus on any uncrowded slide at 10 X magnification.

B. Check centration of the stage and adjust with stage centering screws. If the stage has universal centration then use the objective centering screws to center the 10 X objective.

C. Close field diaphragm located on the base of the stand.

D. Close the aperture diaphragm located on the base of the stand.

E. Center the field diaphragm image using the centering screws on the substage condenser.

F. Open the field diaphragm placing the iris leaf edges on the edge of the field of view.

G. Insert the Bertrand lens and open the aperature diaphragm; focus the filament image by sliding the lamp housing back and forth in its mount.

H. Center the filament image adjusting the lamp housing centering screws.

I. Close the aperature diaphragm, it should appear close to center but does not have to be in sharp focus.

J. Remove the Bertrand lens and adjust the aperature diaphragm for optimum contrast.

K. Adjust the remaining objectives centering each individually to the reference objective. Repeat A - K until each objective is centered.

L. Insert the analyzer at the 0 degree position, adjust the substage polarizer to achieve maximum cancellation of transmitted light.

M. Check the alignment of the reticle crosshairs which shoul be coincident with the polarizer.

N. Insert the 530 nm, first order red, compensator plate 45 degrees to the polarizing direction. The color should agree with the first order red located on the Michel-Levy chart.

2.) Refractive index fluid calibration

A. Refractive index fluids are calibrated against NBS standard reference minerals once each month.

B. Refractive index fluids are calibrated by use of a refractometer once each week.

Asbestos Training Program

1.) The analyst is given three months of on the job training. During this time the new analyst works and trains along side of a experienced analyst.

2.) After the new analyst is trained he or she is sent to the McCrone Research Institute for the Identification of Asbestos by PLM course.

3.) After this training course the analyst is observed to insure proper technique.

4.) All samples are repeated by another analyst to insure proper results.

VI. ANALYTICAL PROCEDURES

Each analytical method used for the RCRA program is taken from:

1) Test Methods for Evaluating Solid Waste - SW 846 , Office of Solid Waste and Emergency Response, U.S. EPA, 3rd Edition, 1986.

2) Methods for Chemical Analysis of Water and Wastes , U.S.E.P.A. March 1979.

Alternate methods developed internally must be demonstrated to be of equal or greater performance than the existing method referenced in 40 CFR Part 261,264, or 265 and published in SW846.

The following requirements must be achieved before an alternate test method is used:

a) A complete procedure for the test method must be written, including all equipment used for the method.

b) A description of the matrices for which the method will be used.

c) Comparative results of the proposed method with the existing method must be given that display supportive information for the proposed test method.

d) Some assessment of the interferences which may prohibit the proposed test method must be included.

e) A description of the required quality control practices to be used to monitor the proposed test method.

This information will be reviewed by the supervisor and Quality Assurance Coordinator and if acceptable will be formally submitted to the U.S. EPA for petition as an alternative test method.

3) Environmental Protection Agency, " Interim Method for the Determination of Asbestos in Bulk Insulation Samples". E.P.A. 600/m4-82-020, Dec. 1982.

4) W.C. McCrone, The Asbestos Particle Atlas, AnnArbor Science Publishers Inc.,Michigan 48106, co 1980.

5) W.C. Dixon, Application of Optical Microscopy in Analysis of Asbestos and Quartz, pg 2-41.,Analytical Techniques in Occupational Health Chemistry, American Chemical Society, Washington, D.C., co 1980.

VII. DATA REDUCTION, VALIDATION, AND REPORTING

Raw data from the Inorganics lab is stored in approved laboratory books, with numbered pages. Organics data is stored on strip charts, or computer print-outs. Quantitation results and Total Ion Chromatograms are printed and archived.

Following reduction of Raw Data, results are transferred to Laboratory Work Sheets and the computerized laboratory management system. Analyst's initials, page reference and analysis data are also recorded. It is the responsibility of the analyst to inspect the Work Sheet and validate that the correct value has been reported.

When completed, a laboratory work sheet is reviewed by the supervisor in charge of that area. Data is reviewed for completeness, the "reasonableness" of the data and accuracy of reporting levels. Once this review is accomplished, the results are printed. Once printed, the report is again reviewed for completeness and accuracy. A signed report is submitted to the Client. A copy of the report is filed, with all supporting laboratory work sheets and is archived for at least three years.

Reporting of Significant Figures

When reporting data, the guidelines established in the Handbook for Analytical Quality Control in Water and Wastewater Laboratories, USEPA, (June, 1972) are followed. The term "significant figure" is used, sometimes loosely, to describe a judgement of the reportable digits in a result. When the judgement is not soundly based, meaningful digits are lost or meaningless digits are reported. On the other hand, proper use of significant figures gives an indication of the reliability of the analytical method used.

The following discussion describes the process of retention of significant figures.

A number is an expression of quantity. A figure or digit is any of the characters 0,1,2,3,4,5,6,7,8,9, which, alone or in combination, serve to express a number. A significant figure is a digit that denotes the amount of the quantity in the particular decimal place in which it stands. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit is in doubt. For example, if a value is reported as 18.8 mg/1, the 18 must be firm while the 0.8 is uncertain, but, presumably better than one of the values 0.7 or 0.9 would be.

The number zero may or not be a significant figure depending on the situation.

Final zeros after a decimal point are always meant to be significant figures. For example, to the nearest milligram, 9.8g is reported as 9.800g.

Zeros before a decimal point with nonzero digits preceeding them are significant. For example, in the number 209, the zero is significant. With no preceeding nonzero digit, a zero before the decimal point is not significant.

If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point. As example, in the number 0.004, the zeros are not significant.

Final zeros in a whole number may or may not be significant. In a conductivity measurement of 1,000 umho/cm, there is no implication by convention that the conductivity is 1000 +/- 1 umho. Rather, the zeros only indicate the magnitude of the number.

A good measure of the significance of one or more zeros interspersed in a number is to determine whether the zeros can be dropped by expressing the number in exponential form. If they can, the zeros may not be significant. For example, no zeros can be dropped when expressing a weight of 100.08 g in exponential form; therefore the zeros are significant. However, a weight of 0.0008 g can be expressed in exponential form as 8 x 10-4 g, so the zeros are not significant. Significant figures reflect the limits in accuracy of the particular method of analysis. It must be decided whether the number of significant digits obtained for resulting values is sufficient for interpretation purposes. If not, there is little that can be done within the limits of the given laboratory operations to improve these values. If more significant figures are needed, a further improvement in method or selection of another method will be required.

Once the number of significant figures obtainable from a type of analysis is established, data resulting from such analyses are reduced according to set roles for rounding off.

Rounding Off Numbers

Rounding off of numbers is a necessary operation in all analytical areas. It is automatically applied by the limits of measurement of every instrument and all glassware. However, when it is applied in chemical calculations incorrectly or prematurely, it can adversely affect the final results. Rounding off should be applied only as described in the following sections.

Rounding Off Rules

If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

The question of significant figures also arises in reading an analog instrument, i.e., analog meter, mercury-in-glass thermometer, peak heights, et cetera. Generally, all -- but the last -- digits are known with certainty. There is, however, uncertainty in the last digit. For purposes of this lab, the limit of uncertainty will be +/- 3, that is, a range of 6. If the last digit is not known within this range, one (1) significant digit should be dropped.

VIII. INTERNAL QUALITY CONTROL CHECKS

Specific Quality Control samples are to be performed for each analytical method.

For Trace Metals these are:

1) Check Standard
2) Calibration Blank
3) Laboratory Control Sample
4) Preparation Blank
5) Sample Duplicates
6) Sample Spikes

The acceptance criteria for each of these Q.C. checks are given in the Calibration Procedures and Frequency - Trace Metals. The corrective action for these checks are also stated in that section.

For Inorganics these are:

1) Check Standard
2) Calibration Blank
3) Laboratory Control Sample
4) Preparation Blank
5) Sample Duplicates
6) Sample Spikes

The acceptance criteria for each of these Q.C. checks are given in the Calibration Procedures and Frequency - Inorganics. The corrective action for these checks are also stated in that section.

For Organics these are:

1) Continuing Calibration
2) Preparation Blank
3) Matrix Spike/ Matrix Spike Duplicate (MS/MSD)
4) Surrogate Recovery
5) Internal Standards

Calibration Procedures and Frequency - Organics, defines the acceptance limits for each Quality Control sample and the required Corrective Action.

For Asbestos these are:

1) Sample Spikes
2) Sample Duplicates
3) Preparation Blank
4) Laboratory Control Samples

Internal Performance Evaluations will be conducted at the discretion of the Quality Assurance Officer. These evaluations will be conducted without analyst awareness to provide an unbiased assessment of the analyst, method, and instrument performance. The samples used will be reference samples (U.S. EPA, Hach, ERA, NBS) introduced into the laboratory as routine samples.

All bulk asbestos work is repeated by a separate analyst after initial analysis. If any deviations are noted then the sample is repeated by both analysts.

Quarterly system audits will be conducted by the Quality Assurance Officer to ensure that Quality Control checks and corrective actions are being observed by the sections of the laboratory.

Recommendations from those audits will be given to the Laboratory Director and Staff Supervisors.

IX.
PERFORMANCE AND SYSTEM AUDITS

In conjunction with our Drinking Water Certification, the Illinois EPA Quality Assurance Officer will conduct inspection of Daily Analytical and an audit of its performance in the Program every two years.

The laboratory participates in a quarterly NIOSH - PAT (Proficiency Analytical Testing) Program.

Annually, the laboratory participates in a Major Discharger Performance Evaluation Study from the U.S. EPA organized by THE BIONETICS CORPORATION. This is an evaluation of the laboratory performance on parameters of the NPDES Program.

The entire laboratory participates in two Water Supply Performance Evaluation Studies per year and two Water Pollution Performance Evaluation Studies per year. Both of these studies are sponsored by the U.S.E.P.A., EMSL, Cinncinnati, Ohio.

The laboratory participates in a quarterly American Industrial Hygiene Association's (AIHA) proficiency analytical testing (PAT) program for the evaluation of airborne asbestos fibers by phase contrast microscopy.

The laboratory participates in the semiannual National Voluntary Laboratory Accreditation Program (NVLAP) for the evaluation of bulk asbestos containing materials.

The laboratory performs a semiannual round robin evaluation for airborne asbestos fibers and bulk asbestos materials with two other commercial laboratories.

X. PREVENTATIVE MAINTENANCE

Maintenance of the equipment is an important part of laboratory operation. The responsibility of routine care lies with the analysts using the instruments. Instrument maintenance manuals are kept on file in each laboratory for frequent reference. Repairs which cannot be performed are contracted to the manufacturer's servicemen. The analytical balances are checked annually under service contracts.

Calendar of Equipment Maintenance

Instruments	Frequency	Procedure
1. Balances Analytical	with each use monthly daily	check zero check span calibrate 100 g internal weight.
Top Loader	with each use	check zero
Triple Beam	with each use	check zero
All balances	annually	service agreement
2. Conductivity Bridge	daily or with each use	check meter against calibrated capacitor. Deviations >10% are reported.
3. pH Meter	daily or with each use	check linearity by using 2 buffers, if linearty is out of control, electrode is replaced.
Ion Selective Electrode	each use	calibrate
4. BOD Incubator	twice daily	temperature is noted in notebook
5. Walk-in Refrigerator	twice daily	if temperature is erratic, call the service rep.
6. Organics Refrigerator	twice daily	if temperature is erratic, call the service rep.

7.	Milli-Q System	twice daily	replace cartridge when resistance falls below 9 Mohm. Replace tank resistance falls below 50000 ohms.
8.	Drying Oven	twice daily	temperature is noted in notebook
9.	Freezer	twice daily	temperature is noted in notebook
10.	Water Bath	twice daily	temperature is noted in notebook
11.	Pure Water Tests		
	pH	monthly	If any pure water tests are outside the recommended limits, the problem must be identified and corrected. Until this is done another source of water must be used.
	Conductivity	daily	
	Standard Plate	monthly	
	Suitability	annually	
	Trace Metals	annually	
	Chlorine Residual	monthly	
	TOC	monthly	
12.	Atomic Absorption Spectrophotometers 305A and 2380		
	Burner Heads	monthly	Clean slot
	Mixing Chamber	monthly	Wash Chamber Spoiler, Nebulizer
	Drain System	daily	Flush with 100ml DI
	Optics	annually	Wash optics and windows with cotton balls and dilute detergent Blow dry.
13.	Graphite Furnace		
	Windows	monthly	Wash with Alcohol
	Optical Sensor	monthly	Wash with Alcohol

Contact Electrodes	monthly	Check for pitting, excessive wear. Replace as required
Test Program	semi-annually	Check Micro Processor

14. B+L Spec 21

Cuvettes	each use	Wash with chromic
Wavelength	annually	Put documentation in equipment file.

15. GC - ECD

septum	weekly	replace
Inlet liner	weekly	clean & replace as needed
detector	semi-annually	wipe test, clean as needed

16. GC - FID

septum	weekly	replace
Inlet liner	weekly	clean & replace as needed
detector	as needed	clean as needed

17. GC - NPD

septum	weekly	replace
Inlet liner	weekly	clean & replace as needed
detector	as needed	clean as needed

18. GC-MS

septum	weekly	replace
concentrator traps	as needed	replace
vacuum pumps	semi-annually	change
Inlet liners	weekly	clean, replace as needed

19. Microscopes	each use	calibrate to Kohler illumination
	weekly	calibrate phase contrast optics with NPL/HSE test slide
	annually	calibrate polarized light optics with with stage micrometer, clean and replace as needed
20. Asbestos Particle Hoods	annually	replace HEPA filter element
	each use	clean work area with wetting agent

XI. SPECIFIC ROUTINE PROCEDURES USED TO ACCESS DATA PRECISION, ACCURACY AND COMPLETENESS.

A). Correlation Coefficient (sometimes called Coefficient of Variance) - Used to measure acceptability of initial calibration curve.

$$r = \frac{n\,(\Sigma xy) - (\Sigma x)\,(\Sigma y)}{\{[n(\Sigma x^2) - (\Sigma x)^2].5\}\ \{[n(\Sigma y^2) - (\Sigma y)^2].5\}}$$

Correlation Coefficient can be calculated on many hand-held calculators. Before using a particular calculator, the user should first determine that the appropriate algorithm is being used. The following set of test data is suggested:

x	y
1	0.2
2	0.4
3	0.7
4	0.9

coefficient of variance = 0.9965

B. Percent Recovery - is used to measure accuracy.

1. Spike in Lab Pure Water

$$\% \text{ Range} = \frac{\text{Observed}}{\text{Predicted}} \times 100$$

$$\% \text{ Recovery} = \frac{O}{S} \times 100$$

where:

O = observed analyte concentration
S = predicted analyte concentration

2. Spike in sample

$$\% \text{ Recovery} = \frac{(O)\,(Ao) - (N)\,(Ao)}{(S)\,(As)}$$

where:

O = concentration observed for spiked sample
Ao = aliquot size of spiked sample

N = concentration observed from Neat Sample
An = NOTE: An, not Ao, is used in the calculation!

S = concentration of spiking solution
As = aliquot size of spike

NOTE: "O" and "N" are concentrations of sample, not digestate. For this equation to be valid, "O" and "N" must be converted to concentration of analyte in the original sample, "dry weight" or "as received".

Example 1

100 ml aqueous sample is digested, concentrated 4/1 and analyzed for analyte, M. An identical 100 ml sample is spiked and treated similarly. Results are:

	Neat	Spiked
Initial Aliquot (ml)	100	100
Final Aliquot (ml)	25	25
Conc. of Spiking Solution	--	100 ppm
Aliquot of Spike (ml)	0	10 ml
Observed Conc.		
digestate	10 ppm	50 ppm
original sample	2.5 ppm	12.5 ppm

$$\% \text{ Recovery} = \frac{(12.5)(100) - (2.5)(100)}{(100)(10)}$$

$$= 1 \text{ or } 100 \% \text{ Recovery}$$

Example 2

A Solid Sample is analyzed similarly

	Neat	Spiked
Initial Aliquot (gram)	0.95	1.244
Final Aliquot (ml)	25	25
Conc. of Spiking Solution	--	10
Aliquot of Spike (ml)	0	25
Observed Conc.		
Digestate	2.0	10.4
Original Sample	52.632	209.003

$$\% \text{ Recovery} = \frac{(209)\ (1.244) - (52.6)\ (1.244)}{(10)\ (25)}$$

$$= 0.776 \text{ or } 77.6\% \text{ Recovery}$$

C). Relative Percent Difference - used to measure precision. The relative percent difference between two numbers, A and B is defined as:

$$RPD = \frac{|A - B| \times 200}{(A+B)}$$

D). Standard Deviation

$$S = \frac{x^2 - (x)^2/N}{N - 1}^{0.5}$$

$$= \frac{(xi-xave)^2}{N - 1}^{0.5}$$

Note that "N - 1" weighting is used for values of N less than 30. Standard Deviation can be calculated on most hand-held calculators. Analyst must verify that the correct algorithm is being used. The following set of test data is suggested.

13
31
19
21
28
19
16

147

mean = 21.000
standard deviation = 6.40312, "N-1" weighting

E. Relative Standard Deviation - is simply the Standard Deviation, as calculated above, divided by the mean.

$$RSD = \frac{S}{x\ ave}$$

where:

RSD = relative standard deviation
S = standard deviation
x ave = mean average

From the above example:

$$RSD = \frac{6.40312}{21} = 0.304091$$

F. Method of Standard Addition, MSA - is used to calculate concentrations when interferences are affecting the result. Results may be obtained from an actual plot, or may be obtained from most hand-held calculators. The analyst must verify that the calculator is correctly calculating the correlation coefficient, slope and result. The following test data is offered:

Spike	Observed Response
0	40
1	60
2	80
4	120

r = 1.000
Slope = 20.0
X - intercept = -2

XII. CORRECTIVE ACTION

Corrective actions to be taken will depend on the results of quality audit functions found.

Matrix Independent Functions

If a Matrix - Independent audit function fails, then all analyses will stop until the function is brought into control. These Matrix - Independent Functions are as follows:

1) Check Standard
2) Calibration Blank
3) Laboratory Control Sample
4) Preparation Blank

Matrix Dependent Functions

If a Matrix - Dependent audit function fails, the analyis is in question and the analysis repeated once. If failure is received in the reanalysis, values from both analyses are reported and all other sample results associated with the Matrix - Dependent Function flagged. Matrix - Dependent Functions are as follows:

1) Sample Duplicates
2) Sample Spikes
3) Surrogate Recovery

XIII. QUALITY REPORTS TO MANAGEMENT

A. The quality assurance officer is responsible for day-to-day quality assurance. On a weekly basis and for each Analytical Data Package, this officer will audit the Quality Control Summary Sheets. These forms include the following:

Inorganic Analyses
Trace Metals
a. Initial and Continuing Calibration Verification
b. Initial and Continuing Blank Determination
c. Spike Sample Recovery
d. Duplicate Results
e. Detection Limits
f. Standard Addition Results
g. Laboratory Control Results
h. Interference Check Sample

Other Inorganic Parameters
a. Calibration Verification
b. Blank Determination
c. Spike Sample Recovery
d. Duplicate Results
e. Laboratory Control Results

Organics Analyses
a. GC/MS Tuning and Mass Calibration
b. Initial Calibration Check
c. Continuing Calibration Verification
d. Reagent Blank Summary
e. Spike/Spike Duplicate Recovery
f. Surrogate Percent Recovery

B. Inorganics Laboratories
1. Initial and Continuing Calibration Verification.

a. Initial Calibration Verification is the analysis of a mid-range Check Standard (and Blank) to verify that the analytical system is functional. This is done following Initial Calibration and immediately prior to any analyses.

b. Continuing Calibration is simply the continued analysis of a Check Standard (and Calibration Blank). This must be performed after every 10 determinations. It is expected that the Check Standard used in continuing calibration will be the same one used during the Initial Calibration. Identify Check Standard Source in space provided. If several Check Standards are required, place additional data in the next column. Acceptance Limits for Initial and Continuing Verification are the same. A new form should be used with each new site and date.

2. Blanks.

a. Trace Metals, Initial and Continuing Calibration Blanks - are simply the concentration observed from acidified Laboratory Pure Water (ie. reagent blanks). Continuing Blanks are measured after every 10 determinations.

Preparation Blanks are aliquots of Laboratory Pure Water which has been taken through the entire procedural process. Typically, 100 mls lab pure water, with acid, will be concentrated to 25 mls and analyzed. One Preparation Blank is to be analyzed with every 20 samples that are digested.

b. Preparation Blanks are specified as to frequency and use after each Inorganic test method.
Acceptance Limits are detection limits of the test with respect to the sample matrix and size taken for the measurement.

3. Spike Sample Recovery.
Spikes should be in the mid-range of the analytical method and, ideally, be one-half to two times the concentration of the neat sample.

Spiked Samples, one for each matrix type should be analyzed. For certain parameters, ie., Total Dissolved Solids, negative spiking may be acceptable. A negative spike is a dilution of a sample by a specified amount. Calculations are identical to positive spikes.

The results of all spiked samples, regardless of Percent Recovery, are to be reported. Results generated by MSA should be noted on lab sheet with a "S" following the value,ie., 10S.

4. Duplicate Sample Analysis.

One sample of each Matrix type, or one every 10 determinations is to be analyzed in duplicate. Acceptance Limits are measured by Relative Percent Difference and are set at 20%. If results are "less than,(<)" Relative Percent Difference cannot be calculated, and "NC" is reported. Since this is not useful information, efforts should be made to avoid particularly clean samples for duplicate analyses.

5. Current Detection Limits and Lab Control Sample.

Lab Control Samples are spikes into lab pure water and are taken through the entire sample preparation. Acceptance is measured by Percent Recovery. Acceptance Limits are stated on the form and are set at 80-120%. If % Recovery falls outside these limits, investigate for analyst error. Verify Calibration Curve with Check Standard. If no error becomes apparent, the sample(s) associated with the Lab Control Sample must be re-analyzed, beginning with sample preparation. Note, all analyses for this parameter will cease until the non-compliance is resolved.

The Instrument Detection Limit is based on two (2) times the Baseline Noise. The IDL's are performed each quarter for each instrument used. Each required detection limit as specified by contract must be met by at least one instrument that can obtain an IDL at that limit or below. When samples are processed, and the values obtained are less than twice the IDL of the instrument, those samples must be analyzed only by the instrument which meets IDL quarterly criteria with respect to the required detection limit. Samples containing analytes at least two times the determined IDL for a particular instrument may be analyzed by the instrument even if that instrument's IDL was not at or below the required detection limit.

6. Standard Addition Results.

Samples not yielding acceptable Percent Recoveries may be performed by the Method of Standard Additions, MSA. All EP-Tox Metals analysis must be conducted by Method of Standard Additions. Raw data from this procedure are recorded on this form.

7. Interference Check Sample.

When analyzing soil matrices by Inductively Coupled Plasma, an Interference Check Sample (ICS) must be analyzed and recoveries for that sample reported.

DATA FLAGS - INORGANICS & TRACE METALS

NR - Not required

U - Indicates the element was analyzed for, but not detected. Report with a detection limit value (e.g.,10U)

E - Indicates a value estimated or not reported due to the presence of interference.

S - Indicates that value determined was quantified by Method of Standard Addition.

R - Indicates spike sample recovery is not in control limits of 75 - 125 %.

* - Indicates duplicate sample analysis is outside of acceptable limits ± 20% (RPD) Relative Percent Difference.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995.

N.C. - Non-Calculable. Used when either or both values in duplicate sample analysis are below the detection limit.

C. Organics Laboratory

1. GC/MS Tuning and Mass Calibration - Tuning of the Mass Spectrometer is verified daily, or every 12 hours, prior to sample analysis by obtaining the spectra of either DFTPP or 4-BFB. The actual % Relative Abundance is recorded on the appropriate lines as provided. Asterisks are used to flag values that are outside acceptance limits. The date, time of run, operator, and file name are also recorded. The name of the tuning file used to control the MS parameter is indicated by circling the appropriate file name.

2. Initial Calibration Check - Retention times and area counts for all compounds in standard runs are documented along with appropriate run file names. The dates(s) of the Initial Calibration along with instrument identification are also recorded.

3. Continuing Calibration Verification - Continuing Calibration runs are documented. Retention times and % Recoveries for all compounds are recorded along with the date and concentration level. Any recalibration of retention time or response is noted on this form. All recoveries outside acceptance limits are flagged with the appropriate mark.

4. Reagent Blank Summary - Any compounds in the target list detected in a procedural blank above the proposed limits, will be recorded on the appropriate Procedural Blank Summary sheet (ie., 624 or 625). The date of analysis, file, name of run, matrix, concentration detected and suspected source of contamination will all be recorded. Asterisked values will indicate that the detected amounts exceeds acceptance limits.

5. Spike/Spike Duplicate Recovery - All matrix duplicate spike results are recorded. Recovery and Duplicate Recovery data are recorded and any values outside the acceptance limits are flagged with an asterisk. The date the analysis was performed and sample description are also documented.

6. Surrogate Percent Recovery - The Percent Recovery compounds from every sample, standard and blank is recorded. The date of analysis and laboratory sample number is also recorded. Any value outside the acceptance limits is noted with an asterisk.

DATA FLAGS - ORGANIC

U - Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution and for percent moisture.

J - Indicates an estimated value. This flag is used either when estimating a concentration for TICs where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the required detection limit.

C - This flag applies to pesticide results where the identification has been confirmed by GC/MS. Single component pesticides greater than or equal to 10 ng/ul in the final extract shall be confirmed by GC/MS.

B - This flag is used when the analyte is found in the blank as well as the sample. This flag must be used for a TIC as well as for a positively identified TCL compound.

- This flag identifies compounds whose concentrations exceed the calibration range of the instrument for that specific analysis. If one or more compounds have a response greater than full scale, the sample or extract must be diluted and re-analyzed. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form 1 for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate Forms 1. The Form 1 for the diluted sample shall have the "DL" suffix appended to the lab sample number and the client sample number.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is re-analyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample numbers (both lab and client) on the Form 1 for the diluted sample, and all concentration values reported on that Form 1 are flagged with the "D" flag.

ADDENDUM 1

SAMPLE BOTTLE PREPARATION

AND

QUALITY CONTROL

Olive Jars 8 oz. Organics	Method 608	*
1/2 Gallon Amber	Method 608	**
Volatile Vials	Volatiles	5 ppb
	Except for:	
	Methylene Chloride	25 ppb
	Toluene	25 ppb
	Dimethyl Keytone	50 ppb

METHOD 608 ACCEPTANCE LIMITS

	SOIL *	WATER **
alpha-BHC	8 ppb	0.05 ppb
beta-BHC	8 ppb	0.05 ppb
delta-BHC	8 ppb	0.05 ppb
Lindane(gamma-BHC)	8 ppb	0.05 ppb
Heptachlor	8 ppb	0.05 ppb
Aldrin	8 ppb	0.05 ppb
Heptachlor Epoxide	8 ppb	0.05 ppb
Endosulfan I	8 ppb	0.05 ppb
4,4' - DDE	16 ppb	0.10 ppb
Dieldrin	16 ppb	0.10 ppb
4,4' - DDD	16 ppb	0.10 ppb
Endosulfan II	16 ppb	0.10 ppb
4,4' - DDT	16 ppb	0.10 ppb
Endrin	16 ppb	0.10 ppb
Endrin Aldehyde	16 ppb	0.10 ppb
Endosulfan Sulfate	16 ppb	0.10 ppb
Methoxychlor	80 ppb	0.50 ppb
Chlordane	80 ppb	0.50 ppb
Toxaphene	80 ppb	0.50 ppb
Arochlor 1016	160 ppb	1.00 ppb
" 1221	80 ppb	0.50 ppb
" 1232	80 ppb	0.50 ppb
" 1242	80 ppb	0.50 ppb
" 1248	80 ppb	0.50 ppb
" 1254	160 ppb	1.00 ppb
" 1260	160 ppb	1.00 ppb

TOC High Sensivity	TOC	500 ppb
Phenol Wastewater	Phenol	100 ppb
Phenol Groundwater	Phenol	5 ppb
1/2 Gallon Plastic	Specific conductance TOC	20 umho 5 ppm
500 ML Plastic Cylinder Unpreserved	Specific conductance TOC	20 umho 5 ppm
500 ML Plastic Cylinder Preserved (H2S04)	Ammonia	0.5 ppm
Grease & Oil	Grease & Oil	2.0 ppm
PCB Vials	PCB	1.0 ppm
1000 ML. Plastic Cylinder Preserved (NaOH)	Cyanide	10 ppb
1000 ML. Plastic Cylinder Unpreserved	Specific Conductance	20 umho
pH Small Plastic	Specific Conductance	20 umho
TOX	TOX	10 ppb
Quarts E-P Toxicity	Calcium Chromium Iron Zinc	200 ppb 10 ppb 100 ppb 20 ppb

Olive Jars 8 oz. Organics	Method 608	*
1/2 Gallon Amber	Method 608	**
Volatile Vials	Volatiles Except for: Methylene Chloride Toluene Dimethyl Keytone	5 ppb 25 ppb 25 ppb 50 ppb

METHOD 608 ACCEPTANCE LIMITS

	SOIL *	WATER **
alpha-BHC	8 ppb	0.05 ppb
beta-BHC	8 ppb	0.05 ppb
delta-BHC	8 ppb	0.05 ppb
Lindane(gamma-BHC)	8 ppb	0.05 ppb
Heptachlor	8 ppb	0.05 ppb
Aldrin	8 ppb	0.05 ppb
Heptachlor Epoxide	8 ppb	0.05 ppb
Endosulfan I	8 ppb	0.05 ppb
4,4' - DDE	16 ppb	0.10 ppb
Dieldrin	16 ppb	0.10 ppb
4,4' - DDD	16 ppb	0.10 ppb
Endosulfan II	16 ppb	0.10 ppb
4,4' - DDT	16 ppb	0.10 ppb
Endrin	16 ppb	0.10 ppb
Endrin Aldehyde	16 ppb	0.10 ppb
Endosulfan Sulfate	16 ppb	0.10 ppb
Methoxychlor	80 ppb	0.50 ppb
Chlordane	80 ppb	0.50 ppb
Toxaphene	80 ppb	0.50 ppb
Arochlor 1016	160 ppb	1.00 ppb
" 1221	80 ppb	0.50 ppb
" 1232	80 ppb	0.50 ppb
" 1242	80 ppb	0.50 ppb
" 1248	80 ppb	0.50 ppb
" 1254	160 ppb	1.00 ppb
" 1260	160 ppb	1.00 ppb

ADDENDUM 5

Groundwater Monitoring and

Field Sampling Protocol

DAILY ANALYTICAL LABORATORIES

GROUNDWATER MONITORING

FIELD SAMPLING PROTOCOL

1.0 INTRODUCTION

The sampling protocol below details the procedures that will be followed by D/A field personnel when collecting groundwater samples from properly constructed two inch diameter monitoring wells. The sampling plan closely follows the RCRA Groundwater Monitoring Technical Enforcement Guidance Document published by the USEPA, Sept., 1986. Any deviations from standard groundwater sampling procedures will be discussed with and approved by the site coordinator prior to implementation. Field groundwater sampling techniques will be updated as required to comply with current EPA standard practice. Additional site-specific requirements not covered in the sampling plan will be issued as an addendum to the document.

1.1 DEPTH TO WATER MEASUREMENTS

Depth to water for all monitoring wells and peizometers are taken using an electronic depth to water meter. The polyethylene measuring tape is rinsed with lab-pure (ASTM type 1 grade) water and wiped with disposable cloth upon retracting from the well. The stainless steel sensor is also rinsed thoroughly with lab-pure water after every measurement. All measurements are recorded to 0.01 ft. Depth to water (from top of well stick-up) and time of measurement are recorded in the field book. To detect possible siltation in the wells, total well depth measurements are recorded once a year. The volume of water contained in each well is then calculated according to the equation:

$$V = r\,h(7.48)$$

where V = volume of water in gallons, r = radius of well (in ft.), and h = total depth of water column (in ft.). Static water measurements are reported as elevations referenced to mean sea level (MSL) if the appropriate information has been provided by a licensed surveyor.

1.2 DETECTION AND SAMPLING OF IMMISCIBLE LAYERS

A portable photoionization detector (PID) is used to "sniff" those wells in which organic contamination is suspect. Variable lamp energies are available to increase sensitivity for known

contaminants. Generally, the PID is calibrated with a 100 ppm isobutylene standard contained in a leak proof tedlar bag. The use of the PID is not standard practice at those sites where routine monitoring has proven to show no immediate concern for heavy organic contamination.

An interface probe will be used to positively identify and measure the existence of immiscible organic layers. If detected, an immiscible layer will be collected prior to purging the well. Lighter "floating" layers are sampled by lowering a translucent teflon bailer with a bottom check valve to a determined depth. The translucent bailer aids in discriminating between the different layers. Dense, bottom layers are collected using a point source top check valve. Care is taken to slowly lower the bailer into the water and avoid mixing layers with the water column.

1.3 WELL PURGING

Before sampling, all wells are purged to remove stagnant water and ensure representative sampling of the aquifer. In accordance with standard EPA protocol, wells are purged until three (3) volumes of water have been removed, or until dryness. If requested, indicator parameters such as specific conductivity and pH can be monitored during purging (usually per well volume) to document removal of all stagnant water. When introducing the bailer into the wells, care is taken to not "drop" it into the water, avoiding agitation and loss of volatile compounds. If possible, wells will be sampled immediately after purging. Slow recharge wells that are bailed to dryness will be sampled at some point during recovery. It is believed that concentrations of volatile analytes will generally be greatest four (4) hours after purging a well dry.* Every attempt will be made to sample slow recharge wells the same day that they are purged.

1.4 SAMPLE COLLECTION

To reduce the possibility of cross-contamination, sampling proceeds from the least contaminated to the most contaminated wells. Here "contamination" is defined as those wells which have been determined to contain the highest concentrations of the specified parameters.

* Herzog, B.L., Chou, S.J., Valkenburg, J.R. and Griffin, R.A. 1988. Changes in Volatile Organic Chemical Concentrations After Purging Slowly Recovering Wells. Groundwater Monitoring Review, v. 8, no. 4, pp. 93-98.

In most cases, sampling will begin with the upgradient wells and proceed downgradient following the flow of the groundwater. After the initial analysis a detailed chronological table detailing the order in which the wells are sampled can be provided.

Sample water is transferred to containers using a spigot, or bottom emptying device. A special controlled flow bottom emptying device is used to fill volatile organic (VOA) and total organic carbon (TOC) vials and amber glass total organic halogen (TOX) containers. Sample bottles are filled according to the volatilization sensitivity of the parameters in the following order:

1. VOA
2. TOX
3. TOC
4. Extractable Organics
5. Phenol
6. Grease and Oil
7. Radioactivity
8. Inorganics
9. Coliform Bacteria
10. Metals
11. Sample water for field analysis

VOA, TOC, and TOX samples are filled and checked to ensure that no air bubbles are entrapped in the containers. To preserve the limited dissolved oxygen environment characteristic of groundwater, all sample bottles will be completely filled and capped with minimum headspace whenever possible. When filling a preserved bottle care is taken to not allow the sample water to overflow, resulting in loss of the preservative. Also, contact between the sampling equipment (spigot) and preserved sample is avoided. Table 1 is a complete listing of all bottle types and preservatives used. Refer to chapter III in the Quality Assurance Project Plan (QAPP) for sample container preparation (cleaning) and storage.

Parameters which require filtration (metals, inorganic anions, TDS, etc.) are transferred in a 1/2 gallon "shuttle" bottle. A nitrogen pressurized, type 316 stainless steel unit is used for field filtration. The sample is poured into a 1.5L reservoir, and filtered through a glass microfiber pre-filter and a 0.45 micron nylon filter. Sample water is filtered directly into appropriately preserved sample bottles. For metals analysis, sample water is filtered immediately after collection, to reduce possible oxidation and precipitation of the analytes.

Finally, all sample bottles collected at each well are labeled (Fig. 1 is an example of a sample label) and placed in ice packed coolers for transport to the laboratory.

1.5 FIELD ANALYSIS

Temperature, pH, and Specific Conductivity field measurements are taken from the last bottle immediately after filling. pH and Spec. Cond. analysis are done in accordance with methods 9040 and 9050, SW-846, respectively. Instrument probes are not placed into a container designated for lab analysis. Sample water is poured into Nalgene beakers for field measurements. The pH meter is calibrated with 7.00 and 4.00 or 10.00 buffer solutions, and automatically adjusts to 100% ideal Nernstian slope. The Spec. Cond. field meter is calibrated against two (2) KCl reference standards. Calibration for both instruments is checked before every reading against a standard, and re-calibration is performed whenever necessary. All instrument probes and beakers are rinsed thoroughly with lab-pure water after every use.

Other "in situ" measurements (Dissolved Oxygen, Chlorine, Turbitity) are not routinely performed in the field, however if requested they will be incorporated into the sampling plan.

1.6 FIELD DECONTAMINATION

Before arriving on site, all sampling equipment is cleaned and dried at the laboratory. Bailers are scrubbed in soapy water, tap water rinsed, dilute acid (HCl) rinsed, tap rinsed, and thoroughly rinsed with lab-pure water. After use at each well, the teflon bailer is disassembled and thoroughly rinsed with lab-pure water. Solvent (Acetone and/or reagent grade Hexane) rinse will be used when specific organic parameters are of interest, or when the bailer has been introduced into an obviously contaminated well. If solvent rinse is used, the equipment will be blown dry with nitrogen gas, followed by thorough rinsing with lab-pure water. All solvent rinsings are kept in a separate waste bucket and returned to the lab for disposal.

Dilute HCl field rinse is used when inorganic analytes are of interest. The disassembled bailer is first rinsed with lab-pure water, followed by the acid rinse, then thoroughly flushed with a second lab-pure water rinse.

Actual detailed protocol for field cleaning non-dedicated bailers is dependent on site conditions, groundwater quality, etc. The use of any combination of solvents, detergents, and dilute acids will at times be unnecessary, and can possibly even be a source of well contamination. The protocol used and integrity of the non-dedicated teflon bailer is the responsibility of the sampling team leader.

Disposable PVC gloves are worn whenever possible to prevent contamination from handling the bailer. In extremely cold weather situations, rubber gloves will be worn by field personnel. These gloves are scrubbed in soapy water and thoroughly rinsed with lab-pure water after every use. Nylon twine is tied to the top of the bailer and used to raise and lower the bailer in the wells. The twine is discarded after every use, and a new piece is used at every sampling location. At no time is a clean bailer allowed to come in contact with the ground or any potentially contaminated area. If the bailer must be set down, it will be wrapped in a plastic "drop cloth".

The stainless steel filtration unit is also disassembled and thoroughly rinsed with lab-pure water after every use. No acid rinses or abrasive soaps are used to clean the filter unit. Filter papers are used one time and discarded. A field blank, using the rinse water, can be sampled and analyzed to ensure the integrity of the field rinsings, rinse water, and all sample equipment.

1.7 FIELD QUALITY CONTROL

The type of quality control measures provided are dependent on the specific requirements of the groundwater monitoring project. Routine quality control includes the collection of field blanks, trip blanks, equipment blanks, and duplicate samples. Frequency and type of quality control measures will be as requested/required by D/A clientele.

Trip blanks are filled at the laboratory into appropriate bottles, and carried in an insulated cooler throughout the sampling event. Field blanks are filled in a manner emulating sampling procedures (e.g. metals field blanks are filtered through the stainless steel filtration unit into an appropriately preserved bottle).

Refer to chapter XI in the QAPP for relative percent difference (RPD) criteria on duplicate sample results. Trip and field blank results are advisory both to the integrity of the sample collection and laboratory analysis procedures. If an analyte of interest is detected in either of these blanks, the sample will be re-analyzed to further confirm/deny the quantitation.

Whenever possible, analytical measures which may delineate between field or laboratory contamination will be taken (e.g. a metals field blank which has shown a quantifiable iron concentration will be re-analyzed by direct aspiration, omitting the potentially contaminating "digestion" process). In cases where re-analysis has confirmed that laboratory procedures are responsible for field blank contamination, all samples will be re-analyzed for that particular analyte.

It would be impractical to list all potential sources of contamination for all analytes and criteria on which every detectable analyte will be evaluated. Historical field and laboratory (routine Q.C. analysis procedure) blanks have provided us with a "baseline" of recurring blank contaminants. Iron, sodium, and zinc in metals analysis and acetone and methylene chloride in V.O.A. analysis do appear in blanks above the method quantitation limits. Field blank results for every site are reviewed against historical data, as with actual sample results. Refer to the corrective action (chapter XII) in the QAPP for data review/re-analysis criteria.

All field duplicate and blank results are reported to the client with the monitoring well results. It is the client's prerogative to call for a re-sampling based on field Q.C. results.

1.8 FIELD NOTEBOOKS

A waterproof field logbook is used to document every sampling event. The following information is recorded for every sampling point:

1. Depth to water
2. Volume (gallons) of water in well
3. Purge volume
4. Time at bailing/sampling
5. pH, temp., and spec. cond.
6. Sample appearance/odor
7. Additional field observations (unusual recharge rates potential contamination sources, etc.)

Additional notations in the field book include the names of personnel on the project, date of sampling, weather conditions, frequency of field equipment calibration, Q.C. lot numbers of sample containers used, and time of "field blank" collection.

1.9 CHAIN OF CUSTODY FORMS

Chain of custody (C.O.C) forms are completed on site and document the custody of every sample collected. Fig. 2 is an example of a typical C.O.C. form. Generally, one (1) form accompanies each set of samples from a well. Each form identifies the well number and types of bottles contained in the cooler. C.O.C. forms are completed and signed by the sampling team leader.

If samples are to be delivered to the laboratory by any party other than the sampling team, or if at any time the samples will be out of the possession of the sampling team, the coolers will be sealed with evidence tape to ensure that the samples are not altered with. Upon arrival at the laboratory, the C.O.C. is completed by the signature of the lab personnel receiving the samples for log-in procedures.

TABLE 1.
DAILY ANALYTICAL LABORATORIES
GROUNDWATER MONITORING
SAMPLE CONTAINERS AND PRESERVATIVES

Parameter	Container type	Preservative	Volume (ml)
Metals	polyethylene, black phenolic cap; conical polyethylene liner	5 ml. conc. nitric	1000
Mercury	polyethylene, black phenolic cap; liner polyethylene liner	HNO3/K2Cr2O7	500
VOA	(2) 40 ml glass vials, black phenolic open top screw cap, teflon lined septum	refrigerated to 4oC	80
B/N/A extr. organics	1/2 gallon amber glass, teflon lined phenolic cap	refrigerated to 4oC	1900
TOX (quad)	(2) 500 ml amber glass, teflon lined phenolic cap	1.0 ml. conc. sulfuric	1000
TOC	(2) 40 ml amber glass vials, black phenolic open top screw cap, teflon lined septum	0.25 ml. conc. sulfuric	80
TOC (quad)	(2) 250 ml amber glass, teflon lined phenolic cap	0.5 ml. conc. sulfuric	500
Phenol	amber glass, teflon lined phenolic cap	2.0 ml. conc. sulfuric	1000

TABLE 1. CONT'D

Parameter	Container type	Preservative	Volume (ml)
Grease and Oil	32 oz flint glass teflon lined phenolic cap	5.0 ml conc. sulfuric	1000
Cyanide Sulfide	polyethylene, black phenolic cap with conical polyethylene liner	5.0 ml. 6N NaOH	1000
Sulfide (titrimetric)	polyethylene, black phenolic cap with conical polyethylene liner	0.5 ml. 2N zinc acetate	250
Coliform	sterilized glass, rubber lined screw cap	0.5 ml. 10% thiosulfate	150
Gross alpha Gross beta Radium	(2) 1/2 gallon polyethylene, polypropylene cap with poly-ethylene liner	0.5 ml. conc. nitric	3800

TABLE 1. CONT'D

Parameter	Container type	Preservative	Volume (ml)
Nitrate Ammonia(m) COD Phosphate	polyethylene, black phenolic cap with conical polyethylene liner	2.0 ml. conc. sulfuric	250
Sulfate Chloride Fluoride TDS	polyethylene, black phenolic cap with conical polyethylene liner	refrigerated to 4oC	1000
Sample water for field analysis/ pre-filtrate	1/2 gallon polyethylene, polypropylene cap with poly-ethylene liner	N/A	1900

* All sample water for metals, inorganics, and TDS analysis is field filtered into the appropriate bottle type